Final
Sampling and Analysis Plan
Volume II – Quality Assurance Project Plan

OU 1 Remedial Investigation Addendum/ Feasibility Study

Former Schilling Air Force Base
Salina, Kansas

January 12, 2007

Prepared for

U.S. Army Corps of Engineers
Kansas City District

Prepared by

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Appendix C – Field Equipment Manuals

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- *Iron CHEMets® Ferrous Iron Procedure*

Appendix D – Kansas City District Data Quality Evaluation Guidance (CENWK, 2006)

Appendix E – Guidelines for Analysis Notebook Documentation for Screening Samples

*Appendix F – *Draft Work Plan, Former SAFB OU 1, Remedial Investigation/Feasibility Study Addendum* (BMcD, 2006).

*Appendix G – *Draft Sampling and Analysis Plan, Volume I – Field Sampling Plan, OU 1 Remedial Investigation/Feasibility Study Addendum, Former Schilling Air Force Base, Salina, Kansas* (BMcD, 2006)

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LIST OF ACRONYMS AND ABBREVIATIONS

ADR	Automated Data Review
AL	Action Limit
AML	Analytical Management Laboratories
APP	Accident Prevention Plan
BMcD	Burns & McDonnell Engineering Company, Inc.
BLRA	Baseline Risk Assessment Addendum
ºC	Degrees Celsius
CENWK	Kansas City District (USACE)
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
cis-1, 2-DCE	cis-1, 2-Dichloroethene
Chemical QA Branch Lab	Chemical Quality Assurance Branch Laboratory
COC	Chain of Custody
CQAR	Chemical Quality Assurance Report
CVAA	Atomic Absorption
1,1-DCE	1,1-Dichloroethene
DoD	Department of Defense
DQO	Data Quality Objectives
DQCR	Daily Quality Control Reports
DQI	Data Quality Indicator
ECB	Environmental Chemistry Branch
EDMS	Environmental Data Management System
EPS	Environmental Priority Services
ESI	Expanded Site Investigation
FPM	*Field Procedures Manual*
FSM	Field Site Manager
FSP	Field Sampling Plan
FUDS	Formerly Used Defense Site
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectroscopy
HAZWOPER	Hazardous Waste Operations and Emergency Response
HCl	Hydrochoric Acid
HDPE	High Density Polyethylene
HNO_3	Nitric Acid
H_2SO_4	Hydrosulfuric Acid
ICBM	Intercontinental Ballistic Missile
IC	Ion Chromatography
ICP-MS	Inductively Coupled Plasma-Mass Spectrometry
IDL	Instrument Detection Limit
IDQTF	Intergovernmental Data Quality Task Force
IDW	Investigation Derived Waste

LIST OF ACRONYMS AND ABBREVIATIONS (continued)

J	Estimated
KDHE	Kansas Department of Health and Environment
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOD	Limit of Detection
LOQ	Limit of Quantitation
LOX	Liquid Oxygen Plant
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
ME	Marginal Exceedence
MEE	Methane, Ethane, and Ethene
mg/kg	milligrams per kilogram
ml	milliliter
MP	Malcolm Pirnie, Inc.
MPC	Measurement Performance Criteria
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NaOH	Sodium Hydroxide
NELAP	National Environmental Laboratory Accreditation Program
OU 1	Operable Unit 1
OVM	Organic Vapor Meter
PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparibility
PCE	Tetrachloroethene
PE	Performance Evaluation
PID	Photoionization Detector
POC	Point of Contact
PQO	Project Quality Objectives
PQL	Practical Quantitation Limit
PRG	Preliminary Remediation Goal
QA	Quality Assurance
QAP	*Quality Assurance Plan*
QAPP	Quality Assurance Project Plan
QC	Quality Control
QCP	Quality Control Plan
QCSR	Quality Control Summary Report
QL	Quantitation Limit
QSM	Quality Systems Manual

LIST OF ACRONYMS AND ABBREVIATIONS (continued)

R	Rejected / Unusable
RCRA	Resource Conservation Recovery Act
REC	Percent Recovery
RI	Remedial Investigation
RI Addendum/FS	Remedial Investigation Addendum/Feasibility Study
RPD	Relative Percent Difference
SAA	Salina Airport Authority
SAFB	Salina Air Force Base
SAP	Sampling and Analysis Plan
SDWA	Safe Drinking Water Act
SEDD	Staged Electronic Data Deliverable
SI	Site Investigation
SOP	Standard Operating Procedure
SQL	Sample Quantitation Limit
SSHS	Site Safety and Health Supervisor
SSHP	Site Health and Safety Plan
SVOC	Semi Volatile Organic Compounds
TC	Table of Contents
TCE	Trichloroethene
TOC	Total Organic Carbon
U	Undetected
UFP-QAPP	*Uniform Federal Policy for Quality Assurance Project Plans*
µg/kg	micrograms per kilogram
µg/l	micrograms per liter
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency
VOC	Volatile Organic Compound
WP	Work Plan

* * * * *

INTRODUCTION

This *Quality Assurance Project Plan* (QAPP) provides a description of quality assurance (QA) and quality control (QC) procedures for executing a remedial investigation (RI) at Operable Unit 1 (OU 1) at the former Schilling Air Force Base (SAFB) Site, located in Salina, Kansas. The Site is located to the southwest of the city of Salina and is shown on Figure 1-1 in the *Final Work Plan, OU 1 Remedial Investigation Addendum/Feasibility Study (RI Addendum/FS), Former Schilling Air Force Base, Salina Kansas* (Burns and McDonnell Engineering Company, Inc. ([BMcD]), 2007) (WP).

The purpose of the QAPP is to establish the policies, organization, objectives, functional activities, and specific QA and QC activities for managing the project. This QAPP is part of the Sampling and Analysis Plan (SAP) and is intended to be used in conjunction with the WP. Volume I of the SAP includes the *Final Sampling and Analysis Plan, Volume I Field Sampling Plan, OU 1 Remedial Investigation Addendum/Feasibility Study (RI Addendum/FS), Former Schilling Air Force Base, Salina, Kansas* (BMcD, 2007) (FSP) and Volume II of the SAP includes the QAPP. Because the WP and FSP contain relevant information for this QAPP, copies of these reports are provided on compact disks as Appendices F and G, respectively. The scope and format of the QAPP was developed from the Intergovernmental Data Quality Task Force (IDQTF) protocols outlined in *Uniform Federal Policy for QAPP, Parts 1, 2a, and 2b* ([UFP-QAPP] IDQTF, 2005).

Malcolm Pirnie, Inc. (MP) has a contract with the United States Army Corps of Engineers (USACE) – Kansas City District (CENWK) to provide Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) support services at the former SAFB Site. MP has subcontracted BMcD to prepare work plan documents, execute the fieldwork, provide daily quality control reports (DQCRs) and monthly reports, perform data validation, and prepare the RI Report Addendum. Development of the QAPP, as well as laboratory-contracting for the primary laboratory, will be provided by BMcD. Contracting of the QA laboratory will be the responsibility of CENWK.

SITE HISTORY AND DESCRIPTION

The Smoky Hill Army Air Base was built in 1942. The base name was changed to the Smoky Hill Air Force Base in 1946 and to SAFB in 1957. In 1942, the base encompassed 365 acres and house 5,000 military personnel. By 1967, the base had expanded to 4,000 acres.

In 1942, the base served as headquarters for the newly created Army Air Corps Unit, 20th Bomber Command, and became the first operational training base for B-29 bombers. The base remained a bomber base for five years after World War II ended and was deactivated in 1949.

In 1951, the base was reopened for the Korean War. Upon reactivation, several improvements to the facility were made, including a 25-mile long jet fuel pipeline that terminated at a bulk fuel tank farm on the base. Additional improvements were made after the Strategic Air Command took over operational control of the base. These included construction of a training building, a boiler facility, a gas distribution system, and a bulk oil storage system. The base was designated a permanent Air Force installation in July 1953. B-47 bombers were operated from the facility.

In 1960, the bombardment wing was transferred to Forbes AFB at Topeka. A squadron of Atlas F intercontinental ballistic missiles (ICBMs) and Nike surface-to-air missiles was deployed to twelve sites around the SAFB and attached to the SAFB for support. New facilities were constructed for storage and repair of both conventional and nuclear munitions and nuclear warhead maintenance. The SAFB was also upgraded to receive B-52 bombers and KC-135 tankers.

The closure of SAFB was announced in 1964 and the site was transferred to the Salina Airport Authority (SAA) in 1966. SAA has since used much of the property in operating the airfield, renamed the Salina Municipal Airport. The remainder of the former SAFB is currently used for light-to-heavy industrial, aviation, and educational purposes. Many of these entities either lease property from the SAA or have purchased their properties outright. Major landowners/operators at the former SAFB include the Kansas State University at Salina, the Salina Area Vocational Technical School, and Kansas National Guard. Local industries include but are not limited to Tony's Pizza, Schwan's Sales Enterprises, Raytheon (Beechcraft), Flower Aviation, and Moore's Midway Aviation.

More detailed information on the history of the former SAFB Site can be found in the September 2005 *Final Remedial Investigation for Operable Unit One at the Former Schilling Air Force Base, Salina, Kansas* (USACE, 2005).

INVESTIGATION HISTORY

Multiple investigations have been conducted at OU 1 at the SAFB. The major investigations include a Site Investigation (SI) in 1996 (USACE, 1996), a SI in 1998 (USACE, 1998), a RI and Baseline Risk Assessment (BLRA) in 1999 (USACE, 1999), an Expanded Site Investigation (ESI) in 2001 (United States Environmental Protection Agency [USEPA], 2001), and a RI in 2005 (USACE, 2005). Other minor investigations that have been conducted within the OU 1 area include Phase I and Phase II

investigations and indoor air quality investigations. Detailed discussions on these investigations, as well as other investigations conducted at the Site, are provided in WP Section 6.0.

PURPOSE

The purpose of this QAPP is to integrate the technical and QA/QC associated with the groundwater, soil, and sediment sampling performed as part of this RI. The objective of the field investigation activities for the RI Addendum/FS is to provide the data to determine the nature and extent of on- and off-site contamination, to assess potential risks to human health and the environment, to develop/screen remedial alternatives, and to, as needed, design remediation approaches. WP Section 6.0 details the areas investigated during previous investigations and areas that have not previously been investigated, presents any known data gaps, and summarizes the rationale and approach that will be used to fill those data gaps.

ORGANIZATION OF QAPP ELEMENTS

This QAPP has been prepared by BMcD as part of the project planning documents for a RI Addendum of OU 1. This QAPP is Volume II of the SAP and is organized as follows:

Main Body of QAPP:

- Introduction
- UFP-QAPP Format Worksheets and Supporting Documentation
- References

Appendices to QAPP:

- *Appendix A – Analytical Management Laboratories, Inc (AML)
  - 2006 AML *Quality Assurance Plan* (QAP)
  - 2006 AML Standard Operating Procedures (SOP)
  - AML Statistical Limits
  - AML Certifications
  - Performance Test Results
  - AML Statement of Laboratory Self Declaration of Compliance
  - AML Kansas Certification
- Appendix B – Environmental Priority Service, Inc. (EPS), *Groundwater, Soil, and Soil Gas Collection and On-Site Analysis, Field Procedures Manual* (EPS, August 2006) (FPM)
- Appendix C – Field Equipment Manuals
  - *Model 580B Organic Vapor Meter (OVM)/Datalogger Instruction Manual*
  - *YSI® 556 MPS Multi Probe System Operations Manual*
  - *Hach® Portable Turbidimeter Instrument and Procedure Manual*
  - *Iron CHEMets® Ferrous Iron Procedure*

- Appendix D – Kansas City District Data Quality Evaluation Guidance (USACE, 2006)
- Appendix E – Guidelines for Analysis Notebook Documentation for Screening Samples
- *Appendix F – WP
- *Appendix G – SAP Volume I FSP

*Note: These appendices are provided on a single compact disc.

In addition, the QAPP references information that is presented in other project documents that is not repeated here to avoid unnecessary duplication. These documents include:

- *Final Quality Control Plan , Former Schilling Air Force Base, CERCLA Process Support*, Salina, Kansas (BMcD, 2006) (QCP)
- WP (Provided on compact disk as Appendix F)
- FSP (Provided on compact disk as Appendix G)
- *Accident Prevention Plan/ Site Safety and Health Plan, OU 1 Remedial Investigation/Feasibility Study Addendum, Former Schilling Air Force Base, Salina, Kansas* (BMcD, 2007) (APP/SSHP)

* * * * * *

QAPP Worksheet #1
(UFP-QAPP Manual Section 2.1)
Title and Approval Page

Site Name/Project Name: Former SAFB CERCLA Process Support

Site Location: OU 1, Former SAFB, Salina, Kansas

Document Title: ***Sampling and Analysis Plan,***
Volume II - Quality Assurance Project Plan
OU 1 Remedial Investigation Addendum/Feasibility Study
Former Schilling Air Force Base
Salina, Kansas

Lead Organization: **CENWK**

Preparer's Name and Organizational Affiliation: Shauna Lawrence, BMcD

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Investigative Organization's Project QC Coordinator/Date: Sharon Shelton
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Printed Name/Organization: Sharon Shelton, BMcD

Primary Contractor's Project Manager/Date: John L Logigian 1/9/07
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Printed Name/Organization: John Logigian, MP

Approval Signature & Lead Organization's Project Manager/Date: Robyn W. Kiefer 1/9/07
Signature
Printed Name/Organization: Robyn Kiefer, CENWK

Approval Signature/Date: _____
Signature
Printed Name/Title:
Approval Authority:

Approval Signature/Date: _____
Signature
Printed Name/Title:
Approval Authority:

Other Approval Signature/Date: _____
Signature
Printed Name/Title:
Approval Authority:

Document Control Numbering System: See QAPP Cover and Worksheet #3

* * * *

QAPP Worksheet #1
(UFP-QAPP Manual Section 2.1)
Title and Approval Page

Site Name/Project Name: Former SAFB CERCLA Process Support

Site Location: OU 1, Former SAFB, Salina, Kansas

Document Title: ***Sampling and Analysis Plan,***
Volume II - Quality Assurance Project Plan
OU 1 Remedial Investigation Addendum/Feasibility Study
Former Schilling Air Force Base
Salina, Kansas

Lead Organization: **CENWK**

Preparer's Name and Organizational Affiliation: Shauna Lawrence, BMcD

Preparer's Address, Telephone Number, & E-Mail Address: 9400 Ward Pkwy, Kansas City, MO 64114
Shauna Lawrence: (816) 822-3940 / slawrence@burnsmcd.com

Preparation Date (Day/Month/Year): 12/January/2007

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Printed Name/Organization: Tracy Cooley, BMcD

Investigative Organization's Project QC Coordinator/Date:_____
Signature
Printed Name/Organization: Sharon Shelton, BMcD

Primary Contractor's Project Manager/Date:_____
Signature
Printed Name/Organization: John Logigian, MP

Approval Signature & Lead Organization's Project Manager/Date:_____
Signature
Printed Name/Organization: Robyn Kiefer, CENWK

Approval Signature/Date:_____
Signature
Printed Name/Title:
Approval Authority:

Approval Signature/Date:_____
Signature
Printed Name/Title:
Approval Authority:

Other Approval Signature/Date:_____
Signature
Printed Name/Title:
Approval Authority:

Document Control Numbering System: See QAPP Cover and Worksheet #3

* * * *

QAPP Worksheet #2
(UFP-QAPP Manual Section 2.2.4)
QAPP Identifying Information

Site Name: Former SAFB, Salina, Kansas

Project Name: RI Addendum/FS at OU 1

Site Number/Code: Formerly Used Defense Sites (FUDS) ID# B07KSO256-03

Operable Unit: OU 1

Contractor Name: MP and BMcD

Contract Number: W912DQ-06-D-0006

Contract Title: Former SAFB CERCLA Process Support

Work Assignment Number: Task 7

1. **Identify guidance used to prepare QAPP:** *Uniform Federal Policy for QAPP, Parts 1, 2a, and 2b, Final, Version 1, March 2005* (IDQTF, 2005)

2. **Identify regulatory program:** FUDS that is using the CERCLA Process as a framework for work being performed

3. **Identify approval entity:** CENWK

4. **The QAPP is (select one):** Generic **Project Specific**

5. **List dates of scoping sessions that were held:** April 13, 2006, May 11, 2006, and July 25, 2006

6. **List dates and titles of QAPP documents written for previous site work, if applicable:**

Title	Approval Date
Quality Assurance Project Plan for Long-Term Groundwater Monitoring Former Schilling Air Force Base Salina, Kansas (USACE, 2006)	May 2006

7. **List organizational partners (stakeholders) and connection with lead organization:**
Kansas Department of Health and Environment (KDHE) – Review and Concurrence
USEPA, Region 7 – Review and Concurrence

8. **List data users:** USACE-CENWK, MP, BMcD, KDHE, USEPA-Region 7, and SAA

9. **If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusion below:**

   - Section 5.3 – Streamlining Data Review is not applicable. No streamlining of the data review process is being performed.

QAPP Worksheet #2
QAPP Identifying Information
(continued)

Circle QAPP elements and required information that are not applicable to the project. Provide an explanation in the QAPP.

Referenced documents include:

QCP = *Final QCP, Former Schilling AFB, CERCLA Process Support, Salina, Kansas* (BMcD, 2006)

WP = *Final Work Plan OU 1 Remedial Investigation Addendum/Feasibility Study, Former Schilling Air Force Base, Salina, Kansas* (BMcD, 2007)

SAP = *Final Sampling and Analysis Plan, Volume I – Field Sampling Plan, OU 1 Remedial Investigation Addendum/Feasibility Study, Former Schilling Air Force Base, Salina, Kansas* (BMcD, 2007)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Crosswalk to Referenced Documents	QAPP Worksheet # or Page #	Required Information
Project Management and Objectives			
2.1 Title and Approval Page		#1	- Title and Approval Page
2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.3 QAPP Identifying Information		TC-1 to TC-8 #2 #3	- Table of Contents - QAPP Identifying Information - Document Control Numbering
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet		#3 #4-1 to 4-6	- Distribution List - Project Personnel Sign-Off Sheet
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	See also QCP Section 2.0	#5 #6 #7 #8	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table
2.5 Project Planning/Problem Definition 2.5.1 Project Planning	WP Section 4.0 and associated	#9-1 to 9-4	- Project Scoping Session Documentation (including Data Needs tables)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Crosswalk to Referenced Documents	QAPP Worksheet # or Page #	Required Information
(Scoping) 2.5.2 Problem Definition, Site History, and Background	figures 6-1 through 6-23	#10	- Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site Maps (historical and present)
2.6 Project Quality Objectives (PQOs) and Measurement Performance Criteria (MPC) 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria	WP Sections 4.0 and 5.0	#11 #12-1 to 12-13	- Site-Specific PQOs - MPC Table
2.7 Secondary Data Evaluation	WP Section 6.0	#13	- Sources of Secondary Data and Information - Secondary Data Criteria and Limitations Table
2.8 Project Overview and Schedule 2.8.1 Project Overview 2.8.2 Project Schedule	WP Section 5.0 and 7.0	#14 #15-1 to 15-12 #16	- Summary of Project Tasks - Reference Limits and Evaluation Table - Project Schedule/Timeline Table
Measurement/Data Acquisition			
3.1 Sampling Tasks 3.1.1 Sampling Process Design and Rationale 3.1.2 Sampling Procedures and Requirements 3.1.2.1 Sampling Collection Procedures 3.1.2.2 Sample Containers, Volume, and Preservation 3.1.2.3 Equipment/ Sample Containers Cleaning and Decontamination Procedures 3.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures	FSP Section 5.0 and associated tables, figures, and appendices. See also QAPP Appendices A, B, C, and E	#17 #18 #19 #20 #21 #22	- Sampling Design and Rationale - Sample Location Map - Sampling Locations and Methods/SOP Requirements Table - Analytical Methods/SOP Requirements Table - Field Quality Control Sample Summary Table - Sampling SOPs - Project Sampling SOP References Table - Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Required QAPP Element(s) and Corresponding QAPP Section(s)	Crosswalk to Referenced Documents	QAPP Worksheet # or Page #	Required Information
3.1.2.5 Supply Inspection and Acceptance Procedures 3.1.2.6 Field Documentation Procedures			
3.2 Analytical Tasks 3.2.1 Analytical SOPs 3.2.2 Analytical Instrument Calibration Procedures 3.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures 3.2.4 Analytical Supply Inspection and Acceptance Procedures	See also QAPP Appendix A for AML's QAP and SOPs and Appendix B for EPS' FPM See also QAPP Tables 24-1 through 24-8	#23 #24 #25	- Analytical SOPs - Analytical SOP References Table - Analytical Instrument Calibration Table - Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table
3.3 Sample Collection Documentation, Handling, Tracking, and Custody Procedures 3.3.1 Sample Collection Documentation 3.3.2 Sample Handling and Tracking System 3.3.3 Sample Custody	FSP Sections 7.0 and 8.0 and associated appendices	#26 #27	- Sample Handling Flow Diagram - Sample Collection Documentation Handling, Tracking, and Custody SOPs - Sample Container Identification - Example Chain-of-Custody (COC) Form and Seal
3.4 Quality Control Samples 3.4.1 Sampling QC Samples 3.4.2 Analytical QC Samples		#28-1 to 28-13	- QC Samples Table - Screening/Confirmatory Analysis Decision Tree
3.5 Data Management Tasks 3.5.1 Project Documentation and Records 3.5.2 Data Package Deliverables 3.5.3 Data Reporting Formats 3.5.4 Data Handling and Management 3.5.5 Data Tracking and Control	WP Section 5.0 QCP Section 5.0 See also Appendix A AML QAP	#29 #30	- Project Documents and Records Table - Analytical Services Table - Data Management SOPs

Required QAPP Element(s) and Corresponding QAPP Section(s)	Crosswalk to Referenced Documents	QAPP Worksheet # or Page #	Required Information
Assessment/Oversight			
4.1 Assessments and Response Actions 4.1.1 Planned Assessments 4.1.2 Assessment Findings and Corrective Action Responses		#31 #32	- Assessments and Response Actions - Planned Project Assessments Table - Audit Checklists - Assessment Findings and Corrective Action Responses Table
4.2 QA Management Reports	WP Section 5.0 and 8.0	#33	- QA Management Reports Table
4.3 Final Project Report	WP Section 5.0		
Data Review			
5.1 Overview			
5.2 Data Review Steps 5.2.1 Step I: Verification 5.2.2 Step II: Validation 5.2.2.1 Step IIa Validation Activities 5.2.2.2 Step IIb Validation Activities 5.2.3 Step III: Usability Assessment 5.2.3.1 Data Limitations and Actions from Usability Assessment 5.2.3.2 Activities	See also QAPP Appendices D and E	#34 #35 #36 #37	- Verification (Step I) Process Table - Validation (Steps IIa and IIb) Process Table - Validation (Steps IIa and IIb) Summary Table - Usability Assessment
5.3 Streamlining Data Review 5.3.1 Data Review Steps To Be Streamlined 5.3.2 Criteria for Streamlining Data Review 5.3.3 Amounts and Types of Data Appropriate for Streamlining	Not applicable	Not Applicable	Not applicable. No streamlining of the data review process is being performed.

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QAPP Worksheet #3
(UFP-QAPP Manual Section 2.3.1)
Distribution List

The distribution list for controlled copies of the QAPP is maintained by and updated by BMcD as the QAPP preparation organization. Controlled copies of the QAPP are assigned to personnel within each project organization. Recipients of controlled copies are provided with all revisions to the QAPP and are responsible for removing all outdated materials from circulation within their organization. Personnel responsible for controlled copies of the document are responsible for distributing QAPP updates and revisions to personnel within their organization. Project team members will have access to controlled copies of the document through the project managers listed here. Personnel with access to the QAPP include, but are not limited to: Project Engineer, Project Technical Lead, Project Chemist, QA Officer, QC Coordinator, Field Site Manager (FSM), field team members, and laboratory staff.

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Tracy Cooley	Project Manager	BMcD	816-822-3369	816-822-3494	tcooley@burnsmcd.com	OU1-QAPP-001
John Logigian	Project Manager	MP	914-641-2690	914-641-2455	jlogigian@pirnie.com	OU1-QAPP-002
Robyn Kiefer	Project Manager	CENWK	816-389-3615	816-426-5550	robyn.v.kiefer@usace.army.mil	OU1-QAPP-003
Jorge Jacobs	Project Manager	KDHE	785-296-8801	785-296-4823	jjacobs@kdhe.state.ks.us	OU1-QAPP-004
Ken Rapplean	Project Manager	USEPA	913-551-7769	913-551-7063	rapplean.kenneth@epa.gov	OU1-QAPP-005
Dr.Tenkasi Viswanathan	QA Director	AML	913-829-0101 Ext. 26	913-829-1181	tviswanathan@amlabinc.com	OU1-QAPP-006
Dr. Tony Bednar	Project Manager	Environmental Chemistry Branch (ECB) Laboratory	601-634-3652	601-634-2742	anthony.j.bednar@erdc.usace.army.mil	OU1-QAPP-007
Pat Martin	Project Manager	EPS	785-823-6205	785-823-6412	Epsinc@midkan.net	OU1-QAPP-008

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QAPP Worksheet #4
(UFP-QAPP Manual Section 2.3.2)
Project Personnel Sign-Off Sheet

Worksheet #4-1 CENWK

Key project personnel from each organization should sign copies of this form to indicate that they have read the applicable QAPP sections and will perform the tasks as described. Each organization will forward signed sheets to BMcD for inclusion in the project file.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Robyn Kiefer	Project Manager	816-389-3615		
Kirk Boese	Project Technical Lead	816-389-3558		
Jerry Montgomery	Project Chemist	816-389-3904		

Worksheet #4-2 MP

Key project personnel from each organization should sign copies of this form to indicate that they have read the applicable QAPP sections and will perform the tasks as described. Each organization will forward signed sheets to BMcD for inclusion in the project file.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
John Logigian	Project Manager	914-641-2690		

Worksheet #4-3 BMcD

Key project personnel from each organization should sign copies of this form to indicate that they have read the applicable QAPP sections and will perform the tasks as described. Each organization will forward signed sheets to BMcD for inclusion in the project file.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Tracy Cooley	Project Manager	816-822-3369		
Shauna Lawrence	Project Chemist	816-822-3940		
Sharon Shelton	Project QC Coordinator	816-822-3168		
Ed Lindgren	Technical Lead	816-822-3595		
To Be Assigned	Field Team Members		When assigned, project team members will be required to sign that they have read applicable sections of the QAPP.	Project team members must read applicable sections of the QAPP prior to participating in the project.

Worksheet #4-4 AML

Key project personnel from each organization should sign copies of this form to indicate that they have read the applicable QAPP sections and will perform the tasks as described. Each organization will forward signed sheets to BMcD for inclusion in the project file.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Dr. Tenkasi Viswanathan	QA Director	913-829-0101 x 26		

Worksheet #4-5 ECB Laboratory

Key project personnel from each organization should sign copies of this form to indicate that they have read the applicable QAPP sections and will perform the tasks as described. Each organization will forward signed sheets to BMcD for inclusion in the project file.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Dr. Tony Bednar	Project Manager	601-634-3652		

Worksheet #4-6 EPS

Key project personnel from each organization should sign copies of this form to indicate that they have read the applicable QAPP sections and will perform the tasks as described. Each organization will forward signed sheets to BMcD for inclusion in the project file.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Pat Martin	Project Manager	785-823-6205		

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QAPP Worksheet #5
(UFP-QAPP Manual Section 2.4.1)
Project Organizational Chart

A project organizational chart is presented on Figure 5-1. The QCP further defines the QC teams, roles, and responsibilities within BMcD and MP.

U.S. ARMY CORPS OF ENGINEERS

The CENWK serves as the lead organization for the project. Ms. Robyn Kiefer is the CENWK Project Manager for the project and will serve as the primary point of contact (POC) for all project-related questions. Ms. Kiefer will also serve as the POC with regulatory entities and members of the general public on matters regarding the project.

MALCOLM PIRNIE, INC.

MP has overall responsibility for the project to the CENWK. MP will act as the program administrator and provide independent QC of all submittals. Mr. John Logigian is the MP Project Manager for the RI.

The Project Risk Assessor role for the RI Addendum/FS at OU 1 is also fulfilled by MP. Mr. Richard Califano will serve as the MP Project Risk Assessor.

BURNS & MCDONNELL ENGINEERING COMPANY, INC.

Mr. Tracy Cooley will serve as the BMcD Project Manager. Mr. Cooley will be responsible for complete coordination of the work, including adequate internal controls and review procedures to eliminate conflicts, errors, and to verify technical accuracy. In addition, Mr. Cooley is responsible for overseeing activities involving sampling and performance of audits.

Mr. Ed Lindgren will serve as the Technical Lead for activities conducted by BMcD. Mr. Lindgren will be responsible for day-to-day oversight and direction for all aspects of the planning, execution, analyses, and reporting of the RI Addendum/FS at the site.

Ms. Sharon Shelton will serve as the Project QC Coordinator. Ms. Shelton will serve as the senior reviewer. The Project QC Coordinator will provide technical QC for the planning, analyses, and reporting.

Ms. Shauna Lawrence will serve as the Project Chemist for BMcD. In this role, she will act as both the QAPP preparer and data validator. The Project Chemist will also oversee activities involving laboratory analyses and data validation.

Ms. Martha Hildebrandt will serve as the Project Geologist for BMcD. Ms. Hildebrandt will oversee activities involving geologic characterization and evaluation. Mr. Tom Zychinski will serve as the Project Hydrogeologist for BMcD. In this role, he will oversee activities involving the hydrogeologic testing, evaluation and hydrogeologic modeling.

A BMcD FSM and a Site Safety and Health Supervisor (SSHS) who are experienced in groundwater and soil/sediment sampling for RIs will be designated for each sampling event. The FSM will supervise the field activities relevant to this project. The SSHS will have direct responsibility for site-specific activities and decisions regarding the immediate safety of investigation personnel. The FSM will report to the BMcD Project Manager and the SSHS will report to the BMcD Project Safety and Health Manager.

Mr. Eric Wenger, Industrial Hygienist, will serve as the BMcD Project Safety and Health Manager for this project and will have ultimate responsibility for the health and safety of field personnel.

PRIMARY ANALYTICAL LABORATORY

AML is the contract laboratory for the analysis of groundwater, surface water, floor drain, soil, and sediment samples for the project. In addition to providing sample analysis, AML will also provide sample bottles and appropriate sample preservation needed for sample collection. AML will be contracted by BMcD. The address for AML is listed below.

Analytical Management Laboratories, Inc.
15130 South Keeler
Olathe, Kansas 66062
Phone: (913) 829-0101
POC: Dr. Tenkasi Viswanathan

As indicated in the CENWK Scope of Work, the analytical laboratories performing sample analysis are required to be certified by the National Environmental Laboratory Accreditation Program (NELAP). In addition, it is required to submit water and soil/sediment samples to laboratories that also have declared compliance with the most recent update to the Department of Defense (DoD) Quality Systems Manual (QSM) Final Version 3 (DoD, 2006). AML, the primary analysis laboratory, has declared themselves as DoD QSM compliant and the Self Declaration Form is included in Appendix A. AML's organizational structure and responsibilities, including an organizational chart, are described in the laboratory's QAP (See Appendix A).

QUALITY ASSURANCE LABORATORY

The ECB Laboratory is the QA laboratory for the project. The QA Lab will provide analysis of QA split samples for comparison with samples analyzed by AML. The QA Laboratory Manager will be

responsible to actively support the implementation of the QA Lab QAPP, maintain accurate SOPs and enforce their use in the laboratory, maintain a work environment that emphasizes the importance of data quality, and provide management support. The methodology and equipment used by the QA laboratory will be functionally equivalent to that of AML. Contact information for the QA Lab is as follows:

US Army Corps of Engineers
3909 Halls Ferry Road
Vicksburg, Mississippi 39180
Phone: (601) 634-3652
POC: Dr. Tony Bednar

The QA Lab will be contracted by CENWK. Further discussion of QA Lab operations, procedures, and requirements is beyond the scope of this QAPP, but is available from CENWK upon request. As such, the QA Lab's SOPs and QAPP are not included or referenced in subsequent worksheets.

REGULATORY ORGANIZATIONS

The KDHE and USEPA will provide regulatory review of project documents, and CENWK will seek concurrence from KDHE and USEPA regarding project activities. Mr. Jorge Jacobs will serve as the KDHE Project Manager. Mr. Ken Rapplean will serve as the USEPA Region 7 Project Manager.

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QAPP Worksheet #6
(UFP-QAPP Manual Section 2.4.2)
Communication Pathways

Communication between project team members is important for a successful project. Project communication pathways and procedures for the timing, and modes of communication are outlined in the following text and table. BMcD and MP personnel will communicate primarily with the CENWK Project Manager. Communication with other government entities (USEPA, KDHE, SAA, etc.) will be coordinated by CENWK. Questions from the general public regarding project activities will be directed to the CENWK Project Manager.

If, during the course of field activities, it becomes necessary to vary approved work plans, verbal approval from the CENWK Project Manager will be required. The verbal approval shall be documented by the project team and should be followed by a written approval from the CENWK. The approval prepared by the project team shall evaluate schedule, cost, and quality impacts, and will include justification and rationale for the proposed variances. Minor variances (i.e., changing the order of planned sample collection activities, minor adjustment of the sampling locations, minor adjustment of procedures which are not expected to impact data quality, etc.) can be implemented in the field prior to written approval. Major variances (i.e., addition or deletion of sampling locations, change in sample collection rates, change of analytical method, and/or a change in data acceptance or rejection procedures) require documented verbal approval, at a minimum, prior to implementation. Most major deviations from planned activities will require revision or addendum to the QAPP by BMcD personnel. At a minimum, a technical memorandum will be submitted to the project file that summarizes the nature of the deviation and its anticipated impact to the project.

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (timing, pathways, etc.)
POC with KDHE and USEPA Region 7	CENWK Project Manager	Robyn Kiefer	816-389-3615	Materials and information about the project will go to the regulatory entities through the lead organization.
Project Technical Lead for Lead Organization	CENWK Project Engineer	Kirk Boese	816-389-3558	In the event that the lead organization project manager is not available, the project engineer will serve as a backup POC.

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (timing, pathways, etc.)
Manage all project investigations	MP Project Manager	John Logigian	914-641-2690	John Logigian will be MP's liaison between Robyn Kiefer, CENWK and Tracy Cooley, BMcD
Manage all project investigations	BMcD Project Manager	Tracy Cooley	816-822-3369	Tracy Cooley will be BMcD's liaison to Robyn Kiefer, CENWK and John Logigian, MP
Back-up POC for Investigative Organization	BMcD Technical Lead	Ed Lindgren	816-822-3595	In the event that the investigative organization's project manager is not available, the Technical Lead will serve as a backup point of contact.
Field Adjustments / Changes	BMcD FSM	To Be Assigned		The FSM will notify Tracy Cooley of any field adjustments and reasons for same at the time of occurrence. Tracy Cooley will notify by phone or email John Logigian and Robyn Kiefer within one business day.
Stop Work and Initiation of Corrective Action	BMcD SHSS	To Be Assigned		The SHSS will notify Tracy Cooley as soon as possible of any work stoppages and implemented corrective actions due to health and safety considerations. Tracy Cooley will notify by phone or email John Logigian and Robyn Kiefer within one business day.
Stop Work and Initiation of Corrective Action	BMcD Project Manager	Tracy Cooley	816-822-3369	Tracy Cooley will notify John Logigian and Robyn Kiefer as soon as possible of any work stoppages and implemented corrective actions.
Daily Field QC Reports	BMcD FSM	To Be Assigned		The FSM will submit via fax or email DQCRs to Tracy Cooley within one business day of sampling. Tracy Cooley will forward the DQCRs to John Logigian and Robyn Kiefer via fax or email upon receipt.

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (timing, pathways, etc.)
Reporting Lab Data Quality Issues	AML QA Director	Dr. Tenkasi Viswanathan	913-829-0101 Ext. 26	Any QA/QC problems with the project field samples will be reported by Dr. Viswanathan to the BMcD Project Chemist within 2 business days. The problem, potential impact, and proposed corrective action will be presented to Tracy Cooley for transmittal to John Logigian and Robyn Kiefer.
Field and Analytical and Corrective Action	BMcD Project QC Coordinator	Sharon Shelton	816-822-3168	The need for corrective action for field and analytical problems will be determined by the BMcD Project Coordinator following discussions with the appropriate project technical personnel. Corrective actions will be documented on the DQCR or transmitted verbally or by email to the MP and CENWK Project Managers.
QAPP Revisions	CENWK Project Manager	Robyn Kiefer	816-389-3615	Any major changes to the QAPP must be approved by the CENWK Project Manager before the change can be implemented.

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QAPP Worksheet #7
(UFP-QAPP Manual Section 2.4.3)
Personnel Responsibilities and Qualifications Table

Resumes are located in the offices of each organization and are available upon request. In addition, resumes for AML are provided in the laboratory's QAP, which is included as Appendix A to this QAPP. Responsibilities were also discussed previously on QAPP Worksheet #5.

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Robyn Kiefer	Project Manager	CENWK	Oversees all aspects of the project and acts as liaison with regulatory agencies and general public.	B.S., Petroleum Engineering M.S., Civil Engineering Project Manager Professional 18 years experience
John Logigian	Project Manager	MP	Manages Project – coordinates between lead agency and investigative contractor.	B.S., Biology and Chemistry M.S., Chemical Engineering 10 years experience as an industrial chemical engineer 18 years experience as a remediation engineer and environmental project manager
Tracy Cooley	Project Manager	BMcD	Manages Project – coordinates document preparation, field samplings, and data reporting. Coordinates with primary contractor.	B.S., Geology 21 years experience
Shauna Lawrence	QAPP Preparer Data Reviewer	BMcD	Prepares QAPP Performs data validation	B.S., Biology 9 years experience
Sharon Shelton	Project QC Coordinator	BMcD	Provides technical QC, oversight, and direction for all aspects of the planning, execution, analyses, and reporting of the RI/FS at the site.	B.S., Biology M.S., Environmental Science 12 years experience
Ed Lindgren	Technical Lead	BMcD	Day-to-day oversight related to project activities.	B.S., Geology M.S., Geology 10 years experience

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Eric Wenger	Health and Safety Officer	BMcD	Responsibility for the health and safety of field personnel	B.S., Biology and Education M.S., Industrial Hygiene Certified Industrial Hygienist 16 years experience
To Be Assigned	FSM	BMcD	Supervises field sampling and coordinates all field activities	
Dr. Tenkasi Viswanathan	QA Director	AML	Manages generation and reporting of analytical data.	Ph.D., Analytical Chemistry M.S., Organic Chemistry B.S., Chemistry 34 years experience See QAP, Appendix A

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QAPP Worksheet #8
(UFP-QAPP Manual Section 2.4.4)
Special Personnel Training Requirements Table

Field personnel training records are maintained in the BMcD corporate offices. The minimum health and safety training for the field sampling team is 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) training and annual refreshers. In addition, the FSM will have HAZWOPER 8-Hour Supervisor Training. While no specific professional certification or training is available for collection of water, soil, and sediment samples, at least one member of the field sampling team will have experience with the sample collection techniques presented in the FSP.

Project Function	Specialized Training By Title or Description of Course	Training Provider	Training Date	Personnel / Groups Receiving Training	Personnel Titles / Organizational Affiliation	Location of Training Records / Certificates[1]
Field Sampling	HAZWOPER 40-Hour Initial Training	BMcD	Various[2]	All members of the field sampling team.	All members of the field sampling team – BMcD	BMcD Offices Certificates available upon request
Field Sampling	HAZWOPER 8-Hour Refresher Training	BMcD	Various[2]	All members of the field sampling team.	All members of the field sampling team – BMcD	BMcD Offices Certificates available upon request
Field Site Supervisor	HAZWOPER 8-Hour Supervisor Training	BMcD	Various[2]	FSM	FSM - BMcD	BMcD Offices Certificates available upon request

[1]Documentation of training is maintained by the BMcD Human Resources Department
[2] Training dates are kept in training records that are maintained by the BMcD Human Resources Department.

As indicated in the CENWK Scope of Work, the analytical laboratories performing sample analysis are required to be certified by the NELAP. In addition, laboratories are required to have declared compliance with the most recent update to the DoD QSM Final Version 3 (DoD, 2006). AML, the primary analysis laboratory, has declared themselve as DoD QSM compliant. A copy of the DoD QSM Self Declaration Form in included in Appendix A.

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QAPP Worksheet #9
(UFP-QAPP Manual Section 2.5.1)
Project Scoping Session Participants Sheet

QAPP Worksheet #9-1 April 13, 2006 Scoping Meeting

Project Name: RI Addendum/FS Addendum OU 1 **Projected Date(s) of Sampling:** 1st Quarter 2007 **Project Manager:** Robyn Kiefer, CENWK	**Site Name:** OU 1, Former SAFB **Site Location:** Salina, Kansas

Date of Session: April 13, 2006
Scoping Session Purpose: Schilling CERCLA Support Kick-Off Meeting

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Robyn Kiefer	Project Manager	USACE	816-389-3615	robyn.v.kiefer@usace.army.mil	Project Manager
Kirk Boese	Project Engineer	USACE	816-389-3558	kirk.d.boese@usace.army.mil	Project Engineer
Chuck Williams	Geologist	USACE	816-389-3575	chuck.e.williams@usace.army.mil	Geologist
Julia Kisser	Geologist	USACE	816-389-3874	Julia.k.kisser@usace.army.mil	Geologist
Jerry Montgomery	Chemist	USACE	816-389-3904	jerry.a.montgomery@usace.army.mil	Chemist
Vanessa Bauders	Industrial Hygienist	USACE	816-389-3567	vanessa.j.bauders@usace.army.mil	Technical Lead
David Daniel	Risk Assessor	USACE	816-389-3910	david.r.daniel@usace.army.mil	Risk Assessor
John Logigian	Project Manager	MP	914-641-2690	jlogigian@pirnie.com	Project Manager
Tracy Cooley	Project Manager	BMcD	816-822-3369	tcooley@burnsmcd.com	Project Manager
Ed Lindgren	QC Coordinator	BMcD	816-822-3595	elindgren@burnsmcd.com	Project QC Coordinator
Diana Marquez	Technical Lead	BMcD	816-822-3453	dmarque@burnsmcd.com	Technical Lead

Comments/Decisions: Summary of the vapor intrusion project tasks and iterative approach was discussed. The scope of the vapor intrusion investigation was limited to OU 1, and buildings within the footprint of the 100 micrograms per liter (µg/L) chlorinated solvent groundwater plume. Data submittals and interim data reports were discussed. Jerry Montgomery will be the POC for database / data management issues.

Action Items:

- BMcD to develop a schedule for all OU 1 and OU 2 work.

Consensus Decisions:

- Staged Electronic Data Deliverable (SEDD) will be used as the electronic data reporting format.

QAPP Worksheet #9-2 May 11, 2006 Scoping Meeting

Project Name: RI Addendum/FS OU 1 **Projected Date(s) of Sampling:** 1st Quarter 2007 **Project Manager:** Robyn Kiefer, USACE	**Site Name:** OU 1, Former Schilling AFB **Site Location:** Salina, Kansas

Date of Session: May 11, 2006
Scoping Session Purpose: Vapor Intrusion WP Working Meeting

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Robyn Kiefer	Project Manager	USACE	816-389-3615	robyn.v.kiefer@usace.army.mil	Project Manager
Kirk Boese	Project Engineer	USACE	816-389-3558	kirk.d.boese@usace.army.mil	Project Engineer
Charles Colbert	Industrial Hygienist	USACE	816-389-3895	charles.r.colbert@usace.army.mil	Industrial Hygienist
Amy Darpinian	Chemist	USACE	816-389-3897	amy.f.darpinian @usace.army.mil	Project Chemist
Vanessa Bauders	Industrial Hygienist	USACE	816-389-3567	vanessa.j.bauders@usace.army.mil	Technical Lead
David Daniel	Risk Assessor	USACE	816-389-3910	david.r.daniel@usace.army.mil	Risk Assessor
John Logigian	Project Manager	MP	914-641-2690	jlogigian@pirnie.com	Project Manager
Richard Califano	Risk Assessor	MP	201-388-4307	rcalifano@pirnie.com	Risk Assessor
Hope Nemickas	Risk Assessor	MP	201-398-4356	hnemickas@pirnie.com	Risk Assessor
Tracy Cooley	Project Manager	BMcD	816-822-3369	tcooley@burnsmcd.com	Project Manager
Ed Lindgren	QC Coordinator	BMcD	816-822-3595	elindgren@burnsmcd.com	Project QC Coordinator
Diana Marquez	Technical Lead	BMcD	816-822-3453	dmarque@burnsmcd.com	Technical Lead
Sharon Shelton	Chemist	BMcD	816-822-3168	sshelton@burnsmcd.com	Project Chemist

Comments/Decisions: The majority of topics during this meeting were directed towards the vapor intrusion investigation. However, a few general issues regarding the documents in the associated areas were addressed.

Consensus Decisions:

- Electronic data deliverables will be submitted in SEDD 2a format.
- Controlled copies of the QAPP with unique document control numbers will be submitted to CENWK, MP, BMcD, KDHE, USEPA, and the analytical laboratories (AML and Chemical QA Branch Lab).
- Steps IIa and IIb validation will be provided in a Quality Control Summary Report (QCSR) or similar. Results of Step III usability assessment will be presented in the RI Addendum/FS.

QAPP Worksheet #9-3 July 25, 2006 Scoping Meeting (Morning Session)

Project Name: RI Addendum/FS OU 1 **Projected Date(s) of Sampling:** 1st Quarter 2007 **Project Manager:** Robyn Kiefer, CENWK	**Site Name:** OU 1, Former SAFB **Site Location:** Salina, Kansas

Date of Session: July 25, 2006
Scoping Session Purpose: OU 1 RI Addendum WP

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Robyn Kiefer	Project Manager	USACE	816-389-3615	robyn.v.kiefer@usace.army.mil	Project Manager
Kirk Boese	Project Engineer	USACE	816-389-3558	kirk.d.boese@usace.army.mil	Project Engineer
Frank Bales	Project Engineer	USACE	816-389-3591	francis.e.bales@usace.army.mil	Project Engineer
Andy Gosnell	Project Geologist	USACE	816-389-3871	andrew.s.gosnell@usace.army.mil	Project Geologist
Cathi Sanders	Project Lawyer	USACE	816-389-3726	Not Provided	Project Lawyer
Chuck Williams	Geologist	USACE	816-389-3575	chuck.e.williams@usace.army.mil	Geologist
Tracy Cooley	Project Manager	BMcD	816-822-3369	tcooley@burnsmcd.com	Project Manager
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Ed Lindgren	Technical Lead	BMcD	816-822-3595	elindgren@burnsmcd.com	Technical Lead

Comments/Decisions: The extent of groundwater contamination was discussed. Questions regarding collection of semivolatile organic compounds (SVOCs) within the OU 1 were discussed. The site nomenclature to be used in identifying the study areas was discussed.

Action Items:

- Risk assessors (including MP) will need to get involved in the decision on how many additional sediment and surface water samples would be required for OU 1.
- The process to be used to define the nature and extent of groundwater contamination was reviewed. Suggestions were made that adjustments be made in the direct-push lines west of I-135 and in the northwestern area to better collect data to support the interim remedial action. Additionally, there was a question regarding the number of monitoring wells that would be installed.
- Data requirements from MP anticipated by August 4, 2006.

Consensus Decisions:

- SVOC samples will be collected at the Liquid Oxygen Plant (LOX) and Sewage Treatment Plant areas, but not at Landfill No 2. since no exposure scenario exists at that location.
- When defining areas, the text name (i.e. Landfill No. 2) will be used. The work plan will note that this is an interim nomenclature and subject to modification.
- Day-care center and the construction worker scenarios will be included in the risk-assessment for OU 1.
- Additional data will be collected at the LOX, Sewage Treatment Plant, and day-care facility.

QAPP Worksheet #9-3 July 25, 2006 Scoping Meeting (Morning Session) (continued)

Consensus Decisions:

- Deep sediment samples are needed from Tony's Road ditch and Dry Creek in order to determine if these areas are leaching out and contributing contamination to the groundwater.
- One deep and one shallow bedrock well will be advanced within OU 1. If a source exists at LOX, a second shallow bedrock well will be advanced at that location.
- Preference is for gauging stations on Dry Creek to be installed prior to the beginning of the late-summer/early fall rainy period.

QAPP Worksheet #9-4 July 25, 2006 Scoping Meeting (Afternoon Session)

Project Name: RI Addendum/FS OU 1 **Projected Date(s) of Sampling:** 1st Quarter 2007 **Project Manager:** Robyn Kiefer, CENWK	**Site Name:** OU 1, Former SAFB **Site Location:** Salina, Kansas

Date of Session: July 25, 2006
Scoping Session Purpose: OU 1 RI Addendum Work Plan

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Robyn Kiefer	Project Manager	USACE	816-389-3615	robyn.v.kiefer@usace.army.mil	Project Manager
Kirk Boese	Project Engineer	USACE	816-389-3558	kirk.d.boese@usace.army.mil	Project Engineer
Frank Bales	Project Engineer	USACE	816-389-3591	francis.e.bales@usace.army.mil	Project Engineer
Andy Gosnell	Project Geologist	USACE	816-389-3871	andrew.s.gosnell@usace.army.mil	Project Geologist
Leo Henning	Section Chief Geologist	KDHE	785-296-1914	lhenning@kdhe.state.ks.us	Senior Geologist
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Ken Rapplean	Project Manager	USEPA	913-551-7769	Rapplean.kenneth@epa.gov	Project Manager
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Ed Lindgren	Technical Lead	BMcD	816-822-3595	elindgren@burnsmcd.com	Technical Lead

Comments/Decisions: A review of the OU 1 RI approach was discussed and concerns and questions were addressed.

Action Items:

- Andy Gosnell and/or Frank Bales (CENWK) are to provide the conductivity line data for the WP development.
- BMcD is to ensure that all items discussed in this scoping meeting are incorporated in the WP.

Consensus Decisions:

- Concerns regarding how the groundwater samples would be collected with a direct-push rig were expressed. Consensus was that the field methods for this were premature at this point in the project.
- It was confirmed that the northeastern plume will be addressed first in the field.
- Questions regarding whether surface and subsurface soil samples would be collected at LOX and Sewage Treatment Plant for both volatile organic compounds (VOCs) and SVOCs. This was discussed in the morning session that soil samples for both analyses would be collected at these areas.
- Questions regarding whether the possibility of passive soil-gas methods were considered as an option to delineate possible sources at the LOX and Sewage Treatment Plant areas. Responses included that the areas seemed small enough that this method would not be required and that KDHE will review the predraft work plan for possible use.
- Concerns regarding available information on the location of sanitary sewer lines leading from the LOX were expressed. Leo Henning (KDHE) wants these taken into consideration during work plan development.

QAPP Worksheet #9-4 July 25, 2006 Scoping Meeting (Afternoon Session) (continued)

Consensus Decisions:

- Concerns regarding how long the bedrock wells would be left in place to collect groundwater were addressed that these borings would be left open for days/weeks.
- A "performance standard" was suggested as part of the work plan to address how many transverse transects will be used for plume delineation.
- Questions regarding whether conductivity lines will be run across the site were asked. Conductivity data had been collected at OU 1 and will be available for this investigation. CENWK is to provide this information (see above).
- A two-three week period will be required before monitoring well installation. This time is used to evaluate the direct-push data, propose monitoring well locations, get approval, and mobilize to the site.
- The work plan should be written with "triggers" to drive the various phases of the WP and that these should be present in the RI Addendum WP. By providing these "triggers" in the RI Addendum WP, modifications to the risk assessment could be made if a significant "discovery" is made.

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QAPP Worksheet #10
(UFP-QAPP Manual Section 2.5.2)
Problem Definition

PROBLEM DEFINITION, SITE HISTORY, AND BACKGROUND

A site history and description is provided in the introduction to this QAPP.

The objectives of this RI Addendum/FS for OU 1 at the SAFB are to describe current site conditions, summarize existing data, and outline future investigation and planning activities necessary to assess and address potential threats to human health and the environment associated with OU 1 at the SAFB. This RI Addendum is being conducted to further investigate areas not fully defined in the previous RI and to investigate two areas not previously investigated under the 2005 RI.

Objectives specific to the RI Addendum portion of the investigation, include:

- Quantify the nature and extent of on-site contamination;
- Determine the nature and extent of any off-site contamination;
- Characterize the physical and chemical nature of the site, including fate and transport mechanisms; and
- Obtain information necessary to evaluate remedial alternatives in the FS.

Objectives specific to the FS portion of the investigation include:

- Develop and evaluate remediation alternatives.

A Vapor Intrusion study is also being conducted at SAFB. This study is being performed under a different task number and all documents for this project will be submitted under separate cover.

The problem to be addressed by the project:

See WP Section 4.0. Discussion is provided for the Data Quality Objectives (DQOs) established to clarify the project objectives, define the type of data needed to meet project objectives, and establish limits for the quantity and quality of data needed to support project decisions.

* * * * *

QAPP Worksheet #11
(UFP-QAPP Manual Section 2.6.1)
Project Quality Objectives / Systematic Planning Process Statements

PQOs define the type, quantity, and quality of data that are needed to answer questions and support environmental decision-making. PQOs are presented as both qualitative and quantitative statements. WP Section 4.0 presents a detailed discussion of the DQOs for the investigation.

Who will use the data?

Data users include project team members from CENWK, MP, BMcD, KDHE, USEPA, and SAA.

What will the data be used for?

The data gathered during the RI will be used to describe current site conditions, summarize existing data, and outline future investigation and planning activities necessary to assess and address the potential threats to human health and the environment associated with the OU 1 at SAFB. Further details of the objectives of the investigation are discussed in WP Section 4.0.

What types of data are needed?

The work plan for the OU 1 area supports an iterative field approach for data collection. WP Section 4.0 discusses the data needs and work plan approach for each area in the OU 1 in further detail. In summary, additional data needs for OU 1 include: determining the presence or absence of contamination at the LOX and Sewage Treatment Plant and Landfill areas, characterization of bedrock at the Jet Engine Repair Hangar/Aircraft Wash Area No. 2 and Building 837/Former Building 847 areas, characterization of soil at the Building 837/Former Building 847 areas, the delineation of groundwater contamination in the northeast portion of OU 1, and installation of additional monitoring wells to monitor the plume characteristics and movement

These data are needed to evaluate fate and transport of the contaminants, characterize risk to human health and the environment, and evaluate the need and/or method and design for remediation.

Data needs for the project include the collection of surface soil, subsurface soil, sediment, surface water, and groundwater samples. The soil, sediment, and surface water samples will be selectively analyzed for VOCs, SVOCs, and/or Resource Conservation Recovery Act (RCRA) list of Metals (i.e., arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver). The groundwater samples collected during direct-push field activities will be analyzed on-site using a field GC for selected chlorinated VOCs with 10 percent being sent to an off-site lab for confirmation analysis of VOCs. Upon installation of the monitoring wells, the groundwater samples collected from the new and existing monitoring wells will be

sampled and analyzed for long-term monitoring parameters including VOCs. Twenty percent of these new monitoring wells, which will be selected once the configuration of the monitoring well network is known, will also be sampled for natural attenuation indicator parameters (methane, ethane, ethane [MEE], iron, anions, sulfide, and total organic carbon [TOC]). Table 11-1 presents the VOC, SVOC, RCRA Metals, and natural attenuation indicator parameters being analyzed by the off-site lab for this RI Addendum. FSP Tables 5-2 through 5-6 and Figures 5-1 through 5-8 illustrate these planned sampling analyses and locations.

Matrix?

Soil, sediment, surface water, and groundwater are the matrices of interest.

How "good" do the data need to be in order to support the environmental decision?

Data are being collected to assess the potential for human exposure regarding the groundwater and/or soil pathway. Worksheet #15, Reference Limits and Evaluation Table, presents the VOCs with their associated action levels (ALs) and project practical quantitation limit (PQL) goals. In addition, Worksheet #12, Measurement Performance Criteria MPC Table, presents a summary of the MPC for each of the data quality indicators (DQIs) being used to evaluate precision, accuracy, representativeness, completeness, and comparability (PARCC).

How much data are needed?

Requirements for the soil, sediment, surface water, and groundwater samples vary based on the area being investigated. The definitive number of samples needed for the RI will not be determined until field activities commence due to the iterative approach to the field investigation. The data requirements for the areas planned for field activities are in FSP Tables 5-2 through 5-6. The sampling rationale and approach for each of these areas is discussed in FSP Sections 5.1 through 5.5 and WP Section 6.0.

Where, when, and how should the data be collected/generated?

Samples will be collected from the locations indicated in FSP Section 5.0 and associated FSP Tables 5-2 through 5-6. Associated FSP Figures 5-1 through 5-8 depict these sample locations.
The field activities are currently set to begin in first quarter 2007.

A summary of the project schedule is presented on Worksheet #16, Project Schedule/Timeline Table.

Who will collect and generate the data?

Field sampling activities will be conducted by BMcD personnel.

Groundwater samples collected during direct-push activities will be analyzed on-site using a field GC by EPS, and 10 percent will be sent to an off-site lab (AML) for confirmation analysis of VOCs. All other samples will be submitted to AML for analysis. Split samples will be sent to the QA lab at a 10 percent frequency of the AML samples. Analysis of the QA split samples will be provided by the ECB Laboratory.

How will the data be reported?

Analytical data will be reported by AML in hard copy and electronic format following each sampling event. Hard copy contents are indicated in Worksheet #29, Project Documents and Records Table. The electronic data deliverable will conform to SEDD 2a format.

Analytical results for the QA split samples will be submitted to the CENWK Project Chemist, as ECB Laboratory is contracted for split sample analysis by CENWK.

Project reporting requirements include:

- DQCRs (QCP Section 5.0)
- Monthly Progress Reports (QCP Section 5.0)
- QCSR (WP Section 5.0)
- RI Report Addendum (WP Section 5.0)
- FS Report (WP Section 5.0)
- BLRA (WP Section 5.0)

How will the data be archived?

Field notebooks, chain-of-custody (COC) forms, field forms, disks, tapes, photographic records, and lab reports will be filed and stored at the BMcD offices located in Kansas City, Missouri. Data provided by the analytical laboratories will be archived electronically in SEDD 2a format. After the first six months following completion of the project, these files may be transferred to long-term storage facilities located in Kansas City, Missouri or transferred to CENWK.

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QAPP Worksheet #12
(UFP-QAPP Manual Section 2.6.2)
Measurement Performance Criteria Table

QAPP Worksheet #12-1 VOCs by SW-846 8260B (Soil/Sediment)

Matrix	Soil/Sediment
Analytical Group	VOCs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
Subsurface Soil FSP Section 6.3 Sediment FSP Section 6.5	Method SW-846 8260B SOP O-VOCs-GCMS-RC	Precision – Lab	Relative Percent Difference (RPD) RPD ≤ 30%	Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) Matrix Spike/Matrix Spike Duplicate (MS/MSD)	A
		Precision – Field	See Table 12-1	Field Duplicates	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL Common contaminants < PQL	Method Blanks	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL Common contaminants < PQL	Trip Blanks Rinsate Blanks	S & A
		Accuracy/Bias	See Table 12-11	Surrogates	A
		Accuracy/Bias	See Table 12-3	LCS MS	A
		Sensitivity	PQLs < PQL Goals See Worksheet #15-1	Method Blanks Field Samples Trip Blanks Rinsate Blanks	A

Matrix	Soil/Sediment
Analytical Group	VOCs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
		Completeness – Field	Samples collected from ≥ 90% of planned locations	Data Completeness Check	S
		Completeness – Lab	Acceptable completeness data ≥ 98% and quality data completeness ≥ 80% of analyzed samples	Data validation and usability assessment	A
		Representativeness	Sampling plan followed, samples properly collected and preserved (as required)	Field Completeness Check	S
		Representativeness	Analytical methods adhered to and performed within holding time.	Requested Analysis Review Holding Time Review	A
		Representativeness	Proper corrective actions performed.	Data Completeness/Usability Check	A
		Comparability	See Table 12-1	QA Split Sample	S & A

AML must meet all MPC as specified in the respective SOP. This includes duplicate pairs (LCS/LCSD, MS/MSD, laboratory duplicates, etc) for precision, spike recovery samples (LCS, MS, surrogate, etc) for accuracy, and blanks and PQLs for sensitivity. Completeness calculations are provided in the text section for Worksheets #12-1 through #12-13.

QAPP Worksheet #12-2 VOCs by SW-846 8260B (Water)

Matrix	Water
Analytical Group	VOCs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
Groundwater FSP Section 6.2 Surface Water FSP Section 6.6	Method SW-846 8260B SOP O-VOCs-GCMS-RC	Precision – Lab	RPD ≤ 30%	LCS/LCSD MS/MSD	A
		Precision – Field	See Table 12-2	Field Duplicates	S & A
		Precision – Field (Verification of on-site VOC data)	Linear Regression Comparison (r^2 ≥ 0.70)	Confirmation Sample (off-site)	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL Common contaminants < PQL	Method Blanks	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL Common contaminants < PQL	Trip Blanks Rinsate Blanks	S & A
		Accuracy/Bias	See Table 12-11	Surrogates	A
		Accuracy/Bias	See Table 12-4	LCS MS	A
		Accuracy/Bias/Precision	Accuracy criteria provided with PE sample	Performance Evaluation (PE) Sample	A
		Sensitivity	PQLs < PQL goals See Worksheet #15-2	Method Blanks Field Samples Trip Blanks Rinsate Blanks	A
		Completeness – Field	Samples collected from ≥ 90% of planned locations	Data Completeness Check	S

Matrix	Water
Analytical Group	VOCs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
		Completeness – Lab	Acceptable completeness data ≥ 98% and quality data completeness ≥ 80% of analyzed samples	Data validation and usability assessment	A
		Representativeness	Sampling plan followed, samples properly collected and preserved (as required)	Field Completeness Check	S
		Representativeness	Analytical methods adhered to and performed within holding time.	Requested Analysis Review Holding Time Review	A
		Representativeness	Proper corrective actions performed.	Data Completeness/Usability Check	A
		Comparability	See Table 12-2	QA Split Sample	S & A
AML must meet all MPC as specified in the respective SOP. This includes duplicate pairs (LCS/LCSD, MS/MSD, laboratory duplicates, etc) for precision, spike recovery samples (LCS, MS, surrogate, etc) for accuracy, and blanks and PQLs for sensitivity. Completeness calculations are provided in the text section for Worksheets #12-1 through #12-13.					

QAPP Worksheet #12-3 SVOCs by SW-846 8270D (Soil/Sediment)

Matrix	Soil/Sediment
Analytical Group	SVOCs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
Subsurface Soil FSP Section 6.3 Surface Soil FSP Section 6.4 Sediment FSP Section 6.5	Method SW-846 8270D O-BNA-SVOCs-RD	Precision – Lab	RPD ≤ 30%	LCS/LCSD MS/MSD	A
		Precision – Field	See Table 12-1	Field Duplicates	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL Common laboratory contaminants < PQL	Method Blanks	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL Common laboratory contaminants < PQL	Rinsate Blanks	S & A
		Accuracy/Bias	See Table 12-12	Surrogates	A
		Accuracy/Bias	See Table 12-5	LCS MS	A
		Sensitivity	PQLs < PQL goal See Worksheet #15-3	Method Blanks Field Samples Rinsate Blanks	A
		Completeness – Field	Samples collected from ≥ 90% of planned locations	Data Completeness Check	S
		Completeness – Lab	Acceptable completeness data ≥ 98% and quality data completeness ≥ 80% of analyzed samples	Data validation and usability assessment	A
		Representativeness	Sampling plan followed, samples properly collected and	Field Completeness Check	S

Matrix	Soil/Sediment
Analytical Group	SVOCs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
			preserved (as required)		
		Representativeness	Analytical methods adhered to and performed within holding time.	Requested Analysis Review Holding Time Review	A
		Representativeness	Proper corrective actions performed.	Data Completeness/Usability Check	A
		Comparability	See Table 12-1	QA Split Sample	S & A
AML must meet all MPC as specified in the respective SOP. This includes duplicate pairs (LCS/LCSD, MS/MSD, laboratory duplicates, etc) for precision, spike recovery samples (LCS, MS, surrogate, etc) for accuracy, and blanks and PQLs for sensitivity. Completeness calculations are provided in the text section for Worksheets #12-1 through #12-13.					

QAPP Worksheet #12-4 SVOCs by SW-846 8270D (Water)

Matrix	Water
Analytical Group	SVOCs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
Surface Water FSP Section 6.6	Method SW-846 8270D O-BNA-SVOCs-RD	Precision – Lab	RPD ≤ 30%	LCS/LCSD MS/MSD	A
		Precision – Field	See Table 12-2	Field Duplicates	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL Common laboratory contaminants < PQL	Method Blanks	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL Common laboratory contaminants < PQL	Rinsate Blanks	S & A
		Accuracy/Bias	See Table 12-12	Surrogates	A
		Accuracy/Bias	See Table 12-6	LCS MS	A
		Sensitivity	PQLs < PQL goals See Worksheet #15-4	Method Blanks Field Samples Rinsate Blanks	A
		Completeness – Field	Samples collected from ≥ 90% of planned locations	Data Completeness Check	S
		Completeness – Lab	Acceptable completeness data ≥ 98% and quality data completeness ≥ 80% of analyzed samples	Data validation and usability assessment	A

Matrix	Water
Analytical Group	SVOCs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
		Representativeness	Sampling plan followed, samples properly collected and preserved (as required)	Field Completeness Check	S
		Representativeness	Analytical methods adhered to and performed within holding time.	Requested Analysis Review Holding Time Review	A
		Representativeness	Proper corrective actions performed.	Data Completeness/Usability Check	A
		Comparability	See Table 12-2	QA Split Sample	S & A
AML must meet all MPC as specified in the respective SOP. This includes duplicate pairs (LCS/LCSD, MS/MSD, laboratory duplicates, etc) for precision, spike recovery samples (LCS, MS, surrogate, etc) for accuracy, and blanks and PQLs for sensitivity. Completeness calculations are provided in the text section for Worksheets #12-1 through #12-13.					

QAPP Worksheet #12-5 MEE by RSK-175 (Water)

Matrix	Water
Analytical Group	MEE
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
Groundwater FSP Section 6.2	RSK Method 175 O-MEE-RC	Precision – Lab	RPD ≤ 30%	LCS/LCSD MS/MSD	A
		Precision – Field	See Table 12-2	Field Duplicates	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL Common contaminants < PQL	Method Blanks	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL Common laboratory contaminants < PQL	Trip Blanks Rinsate Blanks	S & A
		Accuracy/Bias	See Table 12-7	LCS MS	A
		Sensitivity	PQLs < PQL goals See Worksheet #15-5	Method Blanks Field Samples Rinsate Blanks	A
		Completeness – Field	Samples collected from ≥ 90% of planned locations	Data Completeness Check	S
		Completeness – Lab	Acceptable completeness data ≥ 98% and quality data completeness ≥ 80% of analyzed samples	Data validation and usability assessment	A
		Representativeness	Sampling plan followed, samples properly collected and preserved (as required)	Field Completeness Check	S

Matrix	Water
Analytical Group	MEE
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
		Representativeness	Analytical methods adhered to and performed within holding time.	Requested Analysis Review Holding Time Review	A
		Representativeness	Proper corrective actions performed.	Data Completeness/Usability Check	A
		Comparability	See Table 12-2	QA Split Sample	S & A
AML must meet all MPC as specified in the respective SOP. This includes duplicate pairs (LCS/LCSD, MS/MSD, laboratory duplicates, etc) for precision, spike recovery samples (LCS, MS, surrogate, etc) for accuracy, and blanks and PQLs for sensitivity. Completeness calculations are provided in the text section for Worksheets #12-1 through #12-13.					

QAPP Worksheet #12-6 Metals by SW-846 6020A (Soil/Sediment)

Matrix	Soil/Sediment
Analytical Group	Metals
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
Subsurface Soil FSP Section 6.3 Surface Soil FSP Section 6.4 Sediment FSP Section 6.5	Method SW-846 6020A I-ICP-MS-Metals-RC	Precision – Lab	RPD ≤ 20%	LCS/LCSD MS/MSD	A
		Precision – Field	See Table 12-1	Field Duplicates	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL	Method Blanks	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL	Rinsate Blanks	S & A
		Accuracy/Bias	See Table 12-8	LCS MS	A
		Sensitivity	PQLs < PQL goals See Worksheet #15-6	Method Blanks Field Samples Rinsate Samples	A
		Completeness – Field	Samples collected from ≥ 90% of planned locations	Data Completeness Check	S
		Completeness – Lab	Acceptable completeness data ≥ 98% and quality data completeness ≥ 80% of analyzed samples	Data validation and usability assessment	A
		Representativeness	Sampling plan followed, samples properly collected and preserved (as required)	Field Completeness Check	S

Matrix	Soil/Sediment
Analytical Group	Metals
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
		Representativeness	Analytical methods adhered to and performed within holding time.	Requested Analysis Review Holding Time Review	A
		Representativeness	Proper corrective actions performed.	Data Completeness/Usability Check	A
		Comparability	See Table 12-1	QA Split Sample	S & A
AML must meet all MPC as specified in the respective SOP. This includes duplicate pairs (LCS/LCSD, MS/MSD, laboratory duplicates, etc) for precision, spike recovery samples (LCS, MS, surrogate, etc) for accuracy, and blanks and PQLs for sensitivity. Completeness calculations are provided in the text section for Worksheets #12-1 through #12-13.					

QAPP Worksheet #12-7 Mercury by SW-846 7471A (Soil/Sediment)

Matrix	Soil/Sediment
Analytical Group	Metals
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
Subsurface Soil FSP Section 6.3 Surface Soil FSP Section 6.4 Sediment FSP Section 6.5	Method SW-846 7471A I-Mercury-CVAA-RD	Precision – Lab	RPD ≤ 20%	LCS/LCSD MS/MSD	A
		Precision – Field	See Table 12-1	Field Duplicates	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL	Method Blanks	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL	Rinsate Blanks	S & A
		Accuracy/Bias	See Table 12-8	LCS MS	A
		Sensitivity	PQLs < PQL goal See Worksheet #15-7	Method Blanks Field Samples Rinsate Blanks	A
		Completeness – Field	Samples collected from ≥ 90% of planned locations	Data Completeness Check	S
		Completeness – Lab	Acceptable completeness data ≥ 98% and quality data completeness ≥ 80% of analyzed samples	Data validation and usability assessment	A
		Representativeness	Sampling plan followed, samples properly collected and preserved (as required)	Field Completeness Check	S

Matrix	Soil/Sediment
Analytical Group	Metals
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
		Representativeness	Analytical methods adhered to and performed within holding time.	Requested Analysis Review Holding Time Review	A
		Representativeness	Proper corrective actions performed.	Data Completeness/Usability Check	A
		Comparability	See Table 12-1	QA Split Sample	S & A
AML must meet all MPC as specified in the respective SOP. This includes duplicate pairs (LCS/LCSD, MS/MSD, laboratory duplicates, etc) for precision, spike recovery samples (LCS, MS, surrogate, etc) for accuracy, and blanks and PQLs for sensitivity. Completeness calculations are provided in the text section for Worksheets #12-1 through #12-13.					

QAPP Worksheet #12-8 Metals by SW-846 6020A (Water)

Matrix	Water
Analytical Group	Metals
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
Groundwater FSP Section 6.2 Surface Water FSP Section 6.6	Method SW-846 6020A I-ICP-MS-Metals-RC	Precision – Lab	RPD ≤ 20%	LCS/LCSD MS/MSD	A
		Precision – Field	See Table 12-2	Field Duplicates	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL	Method Blanks	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL	Rinsate Blanks	S & A
		Accuracy/Bias	See Table 12-9	LCS MS	A
		Sensitivity	PQLs < PQL goal See Worksheet #15-8	Method Blanks Field Samples Rinsate Blanks	A
		Completeness – Field	Samples collected from ≥ 90% of planned locations	Data Completeness Check	S
		Completeness – Lab	Acceptable completeness data ≥ 98% and quality data completeness ≥ 80% of analyzed samples	Data validation and usability assessment	A
		Representativeness	Sampling plan followed, samples properly collected and preserved (as required)	Field Completeness Check	S

Matrix	Water
Analytical Group	Metals
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
		Representativeness	Analytical methods adhered to and performed within holding time.	Requested Analysis Review Holding Time Review	A
		Representativeness	Proper corrective actions performed.	Data Completeness/Usability Check	A
		Comparability	See Table 12-2	QA Split Sample	S & A
AML must meet all MPC as specified in the respective SOP. This includes duplicate pairs (LCS/LCSD, MS/MSD, laboratory duplicates, etc) for precision, spike recovery samples (LCS, MS, surrogate, etc) for accuracy, and blanks and PQLs for sensitivity. Completeness calculations are provided in the text section for Worksheets #12-1 through #12-13.					

QAPP Worksheet #12-9 Mercury by SW-846 7470A (Water)

Matrix	Water
Analytical Group	Metals
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
Groundwater FSP Section 6.2 Surface Water FSP Section 6.6	Method SW-846 7470A I-Mercury-CVAA-RD	Precision – Lab	RPD ≤ 20%	LCS/LCSD MS/MSD	A
		Precision – Field	See Table 12-2	Field Duplicates	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL	Method Blanks	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL	Rinsate Blanks	S & A
		Accuracy/Bias	See Table 12-9	LCS MS	A
		Sensitivity	PQLs < PQL goal See Worksheet #15-9	Method Blanks Field Samples Rinsate Blanks	A
		Completeness – Field	Samples collected from ≥ 90% of planned locations	Data Completeness Check	S
		Completeness – Lab	Acceptable completeness data ≥ 98% and quality data completeness ≥ 80% of analyzed samples	Data validation and usability assessment	A
		Representativeness	Sampling plan followed, samples properly collected and preserved (as required)	Field Completeness Check	S

Matrix	Water
Analytical Group	Metals
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
		Representativeness	Analytical methods adhered to and performed within holding time.	Requested Analysis Review Holding Time Review	A
		Representativeness	Proper corrective actions performed.	Data Completeness/Usability Check	A
		Comparability	See Table 12-2	QA Split Sample	S & A
AML must meet all MPC as specified in the respective SOP. This includes duplicate pairs (LCS/LCSD, MS/MSD, laboratory duplicates, etc) for precision, spike recovery samples (LCS, MS, surrogate, etc) for accuracy, and blanks and PQLs for sensitivity. Completeness calculations are provided in the text section for Worksheets #12-1 through #12-13.					

QAPP Worksheet #12-10 Anions by EPA Method 300.0 (Water)

Matrix	Water
Analytical Group	Anions (Chloride, Nitrate, Nitrite, Sulfate)
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
Groundwater FSP Section 6.2	EPA Method 300.0 I-Anions-IC-RC	Precision – Lab	RPD ≤ 20%	LCS/LCSD MS/MSD	A
		Precision – Field	See Table 12-2	Field Duplicates	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL	Method Blanks	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL	Rinsate Blanks	S & A
		Accuracy/Bias	See Table 12-10	LCS MS	A
		Sensitivity	PQLs < PQL goals See Worksheet #15-10	Method Blanks Field Samples Rinsate Blanks	A
		Completeness – Field	Samples collected from ≥ 90% of planned locations	Data Completeness Check	S
		Completeness – Lab	Acceptable completeness data ≥ 98% and quality data completeness ≥ 80% of analyzed samples	Data validation and usability assessment	A
		Representativeness	Sampling plan followed, samples properly collected and preserved (as required)	Field Completeness Check	S

Matrix	Water
Analytical Group	Anions (Chloride, Nitrate, Nitrite, Sulfate)
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
		Representativeness	Analytical methods adhered to and performed within holding time.	Requested Analysis Review Holding Time Review	A
		Representativeness	Proper corrective actions performed.	Data Completeness/Usability Check	A
		Comparability	See Table 12-2	QA Split Sample	S & A
AML must meet all MPC as specified in the respective SOP. This includes duplicate pairs (LCS/LCSD, MS/MSD, laboratory duplicates, etc) for precision, spike recovery samples (LCS, MS, surrogate, etc) for accuracy, and blanks and PQLs for sensitivity. Completeness calculations are provided in the text section for Worksheets #12-1 through #12-13.					

QAPP Worksheet #12-11 Sulfide by EPA Method 376.1 (Water)

Matrix	Water
Analytical Group	Sulfide
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
Groundwater FSP Section 6.2	EPA Method 376.1 I-Sulfide-Titrimetry-RC	Precision – Lab	RPD ≤ 30%	LCS/LCSD MS/MSD Laboratory Duplicate	A
		Precision – Field	See Table 12-2	Field Duplicates	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL	Method Blanks	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL	Rinsate Blanks	S & A
		Accuracy/Bias	See Table 12-10	LCS MS	A
		Sensitivity	PQLs < PQL goal See Worksheet # 15-11	Method Blanks Field Samples Rinsate Blanks	A
		Completeness – Field	Samples collected from ≥ 90% of planned locations	Data Completeness Check	S
		Completeness – Lab	Acceptable completeness data ≥ 98% and quality data completeness ≥ 80% of analyzed samples	Data validation and usability assessment	A
		Representativeness	Sampling plan followed, samples properly collected and preserved (as required)	Field Completeness Check	S

Matrix	Water
Analytical Group	Sulfide
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
		Representativeness	Analytical methods adhered to and performed within holding time.	Requested Analysis Review Holding Time Review	A
		Representativeness	Proper corrective actions performed.	Data Completeness/Usability Check	A
		Comparability	See Table 12-2	QA Split Sample	S & A
AML must meet all MPC as specified in the respective SOP. This includes duplicate pairs (LCS/LCSD, MS/MSD, laboratory duplicates, etc) for precision, spike recovery samples (LCS, MS, surrogate, etc) for accuracy, and blanks and PQLs for sensitivity. Completeness calculations are provided in the text section for Worksheets #12-1 through #12-13.					

QAPP Worksheet #12-12 TOC by SW-846 9060 (Water)

Matrix	Water
Analytical Group	TOC
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
Groundwater FSP Section 6.2	SW-846 Method 9060 I-TOC-RC	Precision – Lab	RPD ≤ 30%	LCS/LCSD MS/MSD Lab Duplicate	A
		Precision – Field	See Table 12-2	Field Duplicates	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL	Method Blanks	S & A
		Accuracy/Bias – Contamination	No target compounds > ½ PQL	Rinsate Blanks	S & A
		Accuracy/Bias	See Table 12-10	LCS MS	A
		Sensitivity	PQLs < PQL goal See Worksheet #15-12	Method Blanks Field Samples Rinsate Blanks	A
		Completeness – Field	Samples collected from ≥ 90% of planned locations	Data Completeness Check	S
		Completeness – Lab	Acceptable completeness data ≥ 98% and quality data completeness ≥ 80% of analyzed samples	Data validation and usability assessment	A
		Representativeness	Sampling plan followed, samples properly collected and preserved (as required)	Field Completeness Check	S

Matrix	Water
Analytical Group	TOC
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
		Representativeness	Analytical methods adhered to and performed within holding time.	Requested Analysis Review Holding Time Review	A
		Representativeness	Proper corrective actions performed.	Data Completeness/Usability Check	A
		Comparability	See Table 12-2	QA Split Sample	S & A
AML must meet all MPC as specified in the respective SOP. This includes duplicate pairs (LCS/LCSD, MS/MSD, laboratory duplicates, etc) for precision, spike recovery samples (LCS, MS, surrogate, etc) for accuracy, and blanks and PQLs for sensitivity. Completeness calculations are provided in the text section for Worksheets #12-1 through #12-13.					

QAPP Worksheet #12-13 VOCs by On-Site GC Analysis (Water)

Matrix	Water
Analytical Group	VOCs
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S& A)
Groundwater (Direct-Push) FSP Section 6.2	See FPM (Appendix B)	Precision – Onsite Check	RPD ≤ 25 %	Duplicate	S & A
		Precision – Offsite Lab Confirmation	Linear Regression Comparison (r^2 ≥ 0.70)	Confirmation Sample (off-site)	S & A
		Accuracy/Bias – Contamination	No target compounds > PQL	Method Blanks Rinsate Blanks	S & A
		Accuracy/Bias	Linear Regression (r^2 ≥ 0.97)	Initial Calibration Standards	A
		Accuracy/Bias	REC within 75-125 %	Continuing Calibration Standard	A
		Sensitivity	PQLs = Lowest Calibration Standard	Method Blank	A
		Representativeness	Field Procedures Manual adhered to	Requested Analysis Review	A
		Representativeness	Proper corrective actions performed.	Usability Check	A
		Comparability	Linear Regression Comparison (r^2 ≥ 0.70)	Confirmation Sample (off-site)	S & A

Guidelines for on-site GC analysis are presented in *Guidelines for Analysis Notebook Documentation for Screening Samples*, which is provided in Appendix E.
EPS must meet all MPC as specified above, in their FPM, and in the guidelines noted in Appendix E. This includes duplicate pairs for precision, calibration standards for accuracy, and blanks and for sensitivity.

MEASUREMENT PERFORMANCE CRITERIA

MPC have been determined for the soil, sediment, surface water, and groundwater samples. The criteria pertain to DQIs of precision, accuracy/bias, sensitivity, representativeness, comparability, and completeness. The DQIs provide an indication of the quantitative and qualitative degree of data quality. These MPCs were developed based upon the PQOs, including applicable screening levels (see Worksheet #15), project-specific PQLs, laboratory-derived and/or DoD QSM control limits (Tables 12-3 through 12-12), completeness goals, and duplicate, confirmation, and split sample comparisons. Any laboratory-derived performance criteria were reviewed for appropriateness relative to the PQOs and were found to be acceptable. Further discussion regarding the review of the laboratory-derived criteria, including the DoD QSM criteria, is presented with each of the applicable QC samples in the following sections. Discussion regarding the DQIs, QC samples, control limits for each indicator, and potential corrective actions for outliers are provided in the following sections. A copy of CENWK data validation guidance is provided in Appendix D.

Precision

Precision is the level of agreement among individual measurements of the same chemical or physical property. During the data validation process, precision is expressed in terms of RPD. Chemical concentration data obtained from the analysis of LCSD, MSD, laboratory duplicate, field duplicate, and/or QA split samples will be compared to evaluate analytical precision. The RPD is calculated using the following equation:

$$RPD = \frac{|(D_1 - D_2)|}{(D_1 + D_2)/2} \times 100$$

Where:
- RPD = Relative Percent Difference
- D_1 = Original Sample Concentration
- D_2 = Duplicate Sample Concentration

Perfect precision would be indicated by a RPD of 0 percent.

Matrix Spike Duplicates

MS/MSD pairs are performed to assess analytical precision. SW-846 8260B (VOCs), SW-846 8270D (SVOCs), and SW-846 6020A/7470A/7471A (Metals) specify the analysis of a MS/MSD pair in each analytical batch, as sample volume allows. An analytical batch defined as a grouping of up to 20 field samples. Further discussion of the preparation/analysis of the MS/MSD samples is discussed subsequently under Spike Recovery Studies.

The RPD between the MS/MSD pair is compared to precision control limits established by the laboratory. Control limits used by AML for MS/MSD RPDs are provided in Tables 12-3 through 12-10. Any analyte outside precision control limits requires evaluation by the lab. At a minimum, the calculations will be rechecked. If the associated percent recovery(ies) (REC(s)) are outside control limits, the spiked sample will be evaluated as indicated in Appendix D. If the associated spike REC(s) are within control limits, the spiked sample will be qualified as estimated (J) at a minimum. For gross MS/MSD RPD exceedence, all associated samples may be qualified as estimated (J).

Laboratory Duplicates

Laboratory duplicates are performed to assess analytical precision for inorganic analyses or when MS/MSDs are not appropriate. In these cases, a laboratory duplicate will be analyzed in each sample batch, with a sample batch defined as a grouping of up to 20 samples. The laboratory duplicate is obtained from the same container and handled, prepared, and analyzed at the lab using identical techniques as the original sample.

Control limits used by AML for laboratory duplicates, where applicable, are provided in Table 12-10. Any RPD outside of control limits for the lab duplicate requires evaluation by the lab. At a minimum, the calculations will be rechecked. If no errors are found, the lab duplicate should be reprepped and/or reanalyzed. In the event that the secondary analysis is also outside of QC limits, the data will be flagged during data validation (See QAPP Worksheets #35 and #36).

Field Duplicates

Field duplicate sample results will indicate the precision and reproducibility of sample collection and analytical results. Field duplicate samples are obtained for the soil, sediment, and water samples as indicated in FSP Section 5.0 and its associated FSP Tables 5-2 through 5-6 and FSP Figures 5-1 through 5-8. The project goal is to collect a minimum of 10 percent field duplicate samples during the sampling events. The field duplicate samples will be collected in the same manner and analyzed for the same parameters as field samples from the same location. The acceptance criteria for field duplicates are specified on Table 12-1 (soil/sediment) and Table 12-2 (water). It should be noted that field duplicate samples are expected to have greater variability than lab duplicates.

Any result outside of acceptance criteria for the field duplicate requires evaluation. The sample collection method should be verified to determine likely sources of sample non-homogeneity. Additionally, the calculations should be checked for errors and corrected when necessary. If no calculation errors occurred, then the laboratory should be contacted and requested to verify their results. Additionally, any

information the laboratory can give regarding apparent homogeneity of the sample within the sample container should be obtained. If analytical holding times have not been exceeded and sufficient sample volume remains, it may be beneficial to have the laboratory repeat the sample analysis in instances where the field duplicate results are significantly outside of control limits (i.e., RPDs that exceed control limits by more than 50 percent). In some instances, the corrective action will involve flagging the data during data validation (See QAPP Worksheets #35 and #36) or rejection of the results for the original and duplicate sample.

QA Split Samples

QA split sample results also indicate the precision and reproducibility of sample collection and analytical results. QA split samples will be collected for soil, sediment, surface water, and groundwater samples at a 10 percent frequency. During direct-push field activities, QA split groundwater samples will be sent to the QA lab for VOC analysis at 10 percent frequency of the samples sent to AML for off-site confirmation analysis. QA split sampling is performed by collecting two samples from a given location. QA split samples will be collected at the same location and time as the field duplicate (QC) samples are collected. The QC sample is submitted to the primary analytical laboratory, and the second sample (QA split) is submitted to a different laboratory for analysis. The QA split samples will be sent to ECB Laboratory. FSP Tables 5-2 through 5-6 and FSP Figures 5-1 through 5-9 present the planned locations and analyses for the QA split samples.

Confirmation Samples

Confirmation samples will indicate the precision and accuracy of the on-site field analysis and analytical results. The confirmation samples will be collected for direct-push groundwater samples as indicated in FSP Tables and FSP Figures. Collection of confirmation samples will be performed by collecting two samples from a given location. One sample will be analyzed on-site for selected VOCs using a field GC and the second sample will be submitted to an off-site lab for confirmation VOC analysis. The project goal is to collect a minimum of 10 percent confirmation samples during the direct-push sampling activities.

The off-site confirmation sample results will be compared with the results of the on-site sample results utilizing a least squares linear regression analysis. If the measured concentrations span more than one order of magnitude, the data should be log-transformed to standardize variance which is proportional to the magnitude of measurement. The correlation coefficient (r^2) for the results should be 0.70 or greater for the on-site GC analysis data to be considered screening level data.

For correlation coefficient (r^2) less than the 0.70 comparison control limit, all calculations should be checked for errors and corrected when necessary. Instances where the correlation coefficient (r^2) is significantly below 0.70 requires further review by the project team to determine usability of the suspect and adjacent data points for its intended purpose.

Accuracy/Bias

Accuracy measures the bias of a measurement system and may be defined as the degree of agreement between a measurement and its accepted or true value. The accuracy of chemical results is assessed by examining the results of blank samples and spike recovery studies.

Blank Samples

Blank results are used to evaluate whether field or laboratory handling may have contaminated samples and adversely impacted analytical accuracy by causing false positive or high-biased data. The results of these analyses allow an evaluation of whether detections may represent chemicals introduced into the samples during handling, sample shipment, or analytical preparation and analysis.

Blanks are expected to have no detections of target constituents. Any method, rinsate, or trip blank detection that is greater than one-half (1/2) of the constituent's PQL requires evaluation to determine whether sample reanalysis is necessary. Any exception is made for common lab contaminants (acetone, methylene chloride, phthalates, etc.). For these compounds, the blank concentrations should not exceed the PQL. Any sample associated with a blank that fail these criteria shall be reprocessed in a subsequent preparation blank, except when the sample analysis results in a non-detect result for the contaminant. If no sample volume remains for reprocessing, then the results will be reported with appropriate data qualifiers. Results in field samples that are less than five times the corresponding contaminated blank value are generally considered false positives and flagged accordingly during data validation (See Appendix D). The exception to this is for common lab contaminants, in which results for the field samples less than ten times the corresponding contaminated blank receive this qualification. Instances of gross contamination may require reanalysis and/or resampling if the corresponding field samples have similar detections.

Method Blanks

The analytical method for each analysis performed for the investigation specifies the analysis of a method blank in each sample batch, with a sample batch defined as a grouping of up to 20 field samples. For the method blank, a clean matrix is prepared and analyzed in the same manner as the field samples. Any detection in the method blank indicates potential laboratory contamination of the associated field samples in the analytical batch.

Trip Blanks

The laboratory prepares trip blanks and sends them to the field along with the containers for sample collection. Trip blanks are 40-milliliter (ml) VOC vials of reagent-grade water included with all shipments containing VOC and/or MEE samples. Trip blanks are used to evaluate the possibility of contamination during shipping.

Rinsate Blanks

Rinsate blanks are collected in the field when using non-dedicated equipment to evaluate potential cross-contamination between samples caused by residual contamination on sampling equipment. Rinsate blanks are typically analyzed for the same target analytes as the field samples. Rinsate blank are planned to be collected at a minimum 20 percent of the field samples that are collected with non-dedicated equipment. Depending on the amount of non-dedicated equipment used, rinsate blanks will be collected at least once a week but no more than once a day.

PE Sample

One PE sample will be submitted to the primary lab (AML) for analysis of VOCs in groundwater. Acceptable control limits will be provided by Wibby Environmental for evaluation of results provided by AML. For constituents outside the acceptable control limits, AML should re-check calculations at a minimum. Possible reanalysis and further investigation may be requested for constituents grossly outside the control limits.

Spike Recovery Studies

Spike recovery studies are used to evaluate the ability of the laboratory to recover constituents that are intentionally spiked into the samples. Accuracy of spiked samples is expressed as the REC. The REC is calculated using the following equation.

$$REC = \frac{(SSR - SR)}{SA}$$

Where:
REC = Recovery
SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Amount Added

Perfect accuracy is defined as 100 percent REC. The laboratory statistically develops REC acceptance criteria based upon historical data. In addition, the DoD QSM has established accuracy limits for the analyses of soil, sediment, and water matrices using SW-846 8260B (VOCs), SW-846 8270D (SVOCs), and SW-846 6020A/7470A/7471A (Metals). The laboratory compares the mean REC and standard

deviation of their statistically-established control limits for the method (i.e., all the compounds) to the DoD QSM limits to determine their performance relative to DoD QSM requirements. Since the laboratory's limits are tighter than the DoD QSM criteria, the laboratory uses the DoD QSM criteria. In the event that there are no DoD QSM criteria for a given compound, then the laboratory defaults to use of their statistically-derived limits. The DoD QSM also has provision for acceptance of a limited number of sporadic marginal exceedences (MEs) for analyses that contain greater than 11 parameters. MEs must be random, and the ME control limits should not exceed four times the standard deviation around the mean results of control limit studies. The spike recovery control limits, as well as the method-specific ME limits and number of MEs allowed for each method, where applicable, are provided in Tables 12-3 through 12-9.

For the inorganic analyses, AML provided control limits for each analytical method in the respective SOP (Appendix A). These control limits are presented on Table 12-10.

Further discussion regarding the review of the spike recovery study is presented with each of the applicable QC samples in the following sections. An elevated REC indicates potential for high bias in the data; therefore, non-detect results would be considered reliable. A low REC indicates potential for low bias in the data. Instances of significantly low REC (i.e., REC less than 10 percent for organic analyses and REC less than 30 percent for inorganic analyses), indicate the possibility of low bias and/or false negative results.

Surrogates

Surrogates are added to each sample that undergoes organic analyses. Surrogates are compounds that are not normally found in environmental samples that are added (spiked) into field and QC samples and analyzed for REC. Surrogates are utilized to give an indication of the analytical accuracy of the preparation and analysis methods on a per sample basis.

Surrogate RECs are expected to be within control limits (See Table 12-11 [VOC] and Table 12-12 [SVOC]). The DoD QSM has established surrogate control limits for the following analyses in soil, sediment, and water matrices: VOCs by SW-846 8260B and SVOCs by SW-846 8270D. In addition, the laboratory has statistically-derived acceptance limits that may differ from the DoD QSM criteria. In order to be DoD QSM compliant, the laboratory must be able to meet the DoD QSM QC limits. Since the laboratory's limits are tighter than the DoD QSM criteria, the laboratory uses the DoD QSM criteria. In the event that there are no DoD QSM criteria for a given surrogate, then the laboratory defaults to use of their statistically-derived limits. ME limits are not provided for surrogate spikes in the organic analyses, as they are not applicable.

Any surrogate REC outside of control limits requires evaluation by the lab. At a minimum, calculations should be checked for errors and corrected when necessary. If no calculation errors occurred, instrument performance should be verified. If an instrument problem is found, it should be corrected and the samples reanalyzed. If no instrument problem is found, then the sample should be reanalyzed, as applicable, according to method requirements. AML's SOPs O-BNA-SVOC-RD and O-VOCs-GCMS-RC specify that a diluted sample aliquot should be reanalyzed to mitigate any matrix interference. In the event that the secondary analysis is also outside of QC limits, the data will be flagged during data validation (See QAPP Worksheets #35 and #36).

Laboratory Control Sample

Each of the analytical methods used during the investigation requires the analysis of a LCS in each sample batch, with a sample batch defined as a grouping of up to 20 field samples. For the LCS, an interference-free matrix is spiked with known concentrations of target constituents and analyzed. The intent is to measure analytical accuracy of the method in the absence of sample matrix effects. As a measure of accuracy, the results of the LCS are compared against the known analyte concentrations in the spike to determine REC. In some circumstances, the laboratory also prepares and analyzes a LCSD sample. When available, results of the LCS/LCSD pair provide precision information for the batch.

Any LCS REC outside of control limits requires evaluation by the lab. At a minimum, calculations should be checked for errors and corrected when necessary. If no calculation errors occurred, instrument performance should be verified. If an instrument problem is found, it should be corrected and the samples reanalyzed. Depending upon the number and magnitude of compounds with LCS REC failures, corrective action may include reanalysis of only the LCS or reanalysis of all samples within the batch. For VOC and SVOC analyses with analyte lists that contain greater than 11 compounds, a limited number of sporadic MEs are allowed in the DoD QSM prior to batch reanalysis. For the VOC and SVOC analyte lists provided in Tables 12-3 through 12-6, up to three (3) sporadic MEs are allowed for these analyses. In the event that the corrective action analysis is also outside of QC limits, the data will be flagged during data validation (See QAPP Worksheets #35 and #36).

Matrix Spike Sample/Matrix Spike Duplicate

MS and MSD samples are created in the laboratory by adding known concentrations of target analytes into an aliquot of the applicable site matrix prior to sample preparation and analysis. Three samples (original, MS, and MSD) are used. The MS and MSD are spiked with known concentrations of target compounds while the original is not spiked. All three samples are prepared and analyzed independently using the same techniques. MS/MSD samples provide information on matrix interference encountered

during extraction and analysis (i.e., suppression or enhancement of instrument signals) and also provide a measure of the laboratory's ability to analyze an environmental matrix rather than ideal matrix sample. MS samples are principally used to evaluate accuracy by measuring REC of the spiked compounds. When the MS sample is used together with an associated MSD sample, information is obtained on analytical precision.

The minimum frequency for MS/MSD collection is 5 percent of the field samples. Sufficient sample volumes will be submitted to the laboratories such that project-specific MS/MSDs can be analyzed in each batch. Meeting this requirement for analyses with limited holding times may require the collection of additional MS/MSD samples over the course of the investigation. During sampling activities, the FSM or Project Chemist will coordinate with the laboratories to ensure that they are receiving sufficient sample volumes to meet the project-specific MS/MSD requirements.

Any MS/MSD REC outside of control limits requires evaluation by the lab. At a minimum, calculations should be checked for errors and corrected when necessary. The purpose of the corrective action is to distinguish a true matrix effect from an analytical problem. Corrective actions should include one or more of the following:

- Rerunning the extract
- Spiking another sample in the batch
- If the analyte is a naturally-occuring analyte, respiking the sample at a resolvable concentration relative to the sample concentration

For VOC and SVOC analyses with analyte lists that contain greater than 11 compounds, a limited number of sporadic MEs are allowed in the DoD QSM prior to batch reanalysis. For the VOC and SVOC analyte lists provided in Tables 12-3 through 12-6, up to three (3) sporadic MEs are allowed for these analyses. Due to minimal analytes in the metals analysis, no ME are allowed per DoD QSM.

Sensitivity and Practical Quantitation Limits

Sensitivity is the ability of the method to detect target constituents at a level of interest. The PQL is the minimum concentration of a constituent that can be routinely quantified by a lab. The DoD QSM uses the term Limit of Quantitation (LOQ) to describe the PQL, while the UFP QAPP guidance uses the term QL to describe the PQL. In contrast, the method detection limit (MDL) is a statistically derived value that represents the 99 percent confidence level that the reported signal is different from a blank sample. The DoD QSM uses the term Limit of Detection (LOD) to describe the MDL. MDLs are lower values than PQLs. In general, PQLs should be established at least three times the MDL. AML reports detections

between the MDL and PQL as estimated (J-flag). PQLs are often adjusted based upon sample volume and dilution. These adjusted PQLs are referred to as sample quantitation limits (SQLs). Table 12-13 presents a cross-reference to each of these limits and a short definition describing each limit.

PQLs and SQLs are compared to project ALs. ALs are numerical criteria that are used as decision points for the project. The DQOs presented in WP Section 4.0 indicate that any one of several numerical criteria may act as ALs. For purposes of reviewing analytical method PQLs, the ALs selected for soil are the USEPA Region 9 Preliminary Remediation Goals ([PRGs] October 2004) and the ALs used for water are the Safe Drinking Water Act (SDWA) Maximum Contaminant Levels (MCLs). QAPP Worksheet #15 presents a comparison of these screening levels, where applicable, to laboratory MDLs and PQLs for adequacy. Based on an evaluation of uncertainty associated with the RI Addendum analyte list, it was determined that a PQL goal of 1/3 of the AL, which corresponds to recommendations in the UFP-QAPP Manual, was appropriate for most target constituents. For certain "poor performing" analytes, the PQL goal was established at 1/10 of the AL. The "poor performing" analytes as identified in the DoD QSM include:

- Water: 4-nitrophenol, benzoic acid, and phenol
- Soil: 3,3'-dichlorobenzidine, 4-chloroaniline, and benzoic acid

For this evaluation, uncertainty was estimated for each parameter using the LCS acceptance limits. Sensitivity will be assessed by review of the PQLs for non-detect analytes for each method blank and sample versus the PQL goals and ALs on Worksheet #15.

Sensitivity will be assessed for non-detect analytes in method blanks as these samples generally present the "best case scenario" sample. These samples are "clean" samples meaning they are dilution-free and matrix interference is unlikely. Comparison of field sample PQLs to method blank PQLs provides a quick check of lab instruments and/or analytical methods abilities to meet established detection limits, and thus, sensitivity.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is a qualitative parameter that depends upon the proper design of the sampling program and proper laboratory protocols. The following factors impact representativeness: appropriate rationale used to select sampling locations and analytical parameters, correct sample collection techniques

and preservation, use of standard analytical methods, adherence to method holding times, and determination of potential analysis interferences. The representativeness of the data will be determined by:

- Qualitative comparison of actual sampling procedures to those presented in the FSP.
- Quantitative comparison of analytical results for field duplicates and/or field splits to determine parameter variation at a sampling point.
- Invalidating nonrepresentative data or identifying data to be classified as questionable through qualitative or quantitative data validation procedures.
- Review of sample analysis relative to the method holding time.
- Review of sample preservation requirements (temperature blank, applicable sample preservation noted in the respective SOP).

Nonrepresentative or questionable data are data that do not accurately reflect site conditions. If data are determined to be nonrepresentative, they will not be used in subsequent data reduction, validation, and site characterization. If a data point or parameter is determined to be nonrepresentative, the need for additional data collection will be carefully assessed by the BMcD Project Manager in conjunction with MP and CENWK Project Managers.

Comparability

Comparability is a qualitative parameter used to express the confidence with which one data set may be compared to another. Only data with known precision and bias can accurately be compared. Therefore, only data that meets project accuracy and precision criteria will be used for comparability determination. QC data will be provided by the laboratory so end-users can estimate data uncertainty. The data sets may include data generated by laboratories in different phases of a project and/or data generated using different sampling and analysis techniques than those presented in the FSP. Comparability of data is achieved through the use of standard methods to collect and analyze samples, and reporting results in standard units and consistent format. To produce comparable data, the units specified for analytical results obtained during the field investigations will be consistent throughout this project, and standardized analytical methods will be utilized for each parameter.

Comparability can be assessed by collection and submission of split samples to two different laboratories for analysis. QA split samples and confirmation samples are being collected during the RI Addendum

and will be used to assess data comparability. Discussion of these samples is provided in the Precision section of this worksheet.

Completeness

Completeness defines the percentage of measurements judged to be valid measurements. Completeness is assessed for both field and laboratory activities.

Field Completeness

Field completeness is assessed by comparing the number of acceptable samples collected to the number of samples planned for collection, as follows:

$$\% \text{ Field Completeness} = \frac{\text{Number of acceptable samples collected}}{\text{Number of samples planned}} \times 100$$

Acceptable samples include all samples that were properly collected (i.e., no air bubbles present in VOA samples, correct sample temperature and/or preservation, etc.). The field completeness goal for this project is 90 percent. If field completeness falls below 90 percent, the need for additional data collection to meet project objectives will be carefully assessed by the BMcD Project Manager in conjunction with MP and CENWK Project Managers.

Laboratory Completeness

Laboratory completeness for acceptable data is defined as the percentage of acceptable data out of the total amount of data generated. Acceptable data not only includes data which passed all criteria, but also data which may not have passed QC criteria but which had appropriate corrective actions taken. Therefore, rejected data do not adversely effect the analytical completeness calculations if the laboratory has performed appropriate corrective actions and has provided a suitable replacement data point. The acceptable data goal for this project is 98 percent for each individual analytical method. If laboratory completeness falls below 98 percent, the need for additional data collection will be carefully assessed by the BMcD Project Manager in conjunction with MP and CENWK Project Managers.

Laboratory completeness for acceptable data is calculated as follows on a per method and matrix basis:

$$\% \text{ Acceptable Data} = \frac{\text{Number of Compounds with Acceptable Data}}{\text{Number of Compounds Analyzed}} * 100$$

Laboratory completeness for quality data completeness is defined as the percentage of quality data out of the total amount of data generated. Only data that passed all QC criteria as defined in the methods is

considered quality data. Laboratory corrective actions for QC failures and are not taken into consideration when calculating quality data completeness. The laboratory completeness goal for quality data for this project is 80 percent. Quality data completeness is calculated as follows on a per method and matrix basis:

$$\%\ Quality\ Data = \frac{Number\ of\ Compounds\ with\ Quality\ Data}{Number\ of\ Compounds\ Analyzed} * 100$$

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QAPP Worksheet #13
(UFP-QAPP Manual Section 2.7)
Secondary Data Criteria and Limitations Table

This worksheet identifies all secondary data and information that will be used for the project and their originating sources. The use of the secondary data is specified as well as any limitations on their use. WP Section 6.0 discusses a more detailed review of each investigation.

Secondary Data	**Data Source** (originating organization, report title and date)	**Data Generator(s)** (originating organization, data types, data generation / collection dates)	**How Data Will Be Used**	**Limitations on Data Use**
Soil and Groundwater Data	*Final Site Investigation Report, Site Investigation, Former Schilling Air Force Base, Salina, Kansas (USACE, October 1996)*	A site investigation was conducted at areas within OU 1. Soil and groundwater samples were collected and analyzed for SVOCs, VOCs, and/or metals. Results of the investigation were presented in these reports.	To determine the extent and nature of the contamination	See specific area for data use in WP Section 6.0.
	Site Investigation at the Former Schilling Air Force Base, Salina, Kansas (USACE, December 1998)			
	Remedial Investigation at the Former Schilling Air Force Base, Salina, Kansas (USACE, February 1999)			
	Expanded Site Investigation for Schilling (EX) Air Force Base Site, Salina, Kansas, CERCLIS KS0000597922 (USEPA Region 7 Superfund Division, March 2001)			
	Remedial Investigation for Operable Unit One at the Former Schilling Air Force Base, Salina, Kansas (USACE, September 2005)			

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QAPP Worksheet #14
(UFP-QAPP Manual Section 2.8.1)
Summary of Project Tasks

SAMPLING TASKS:

Soil, sediment, surface water, and/or groundwater samples will be collected and analyzed from four areas as part of the RI at SAFB OU 1. These areas include the Sewage Treatment Plant and Landfill area, LOX Plant, Jet Engine Repair Hangar/Aircraft Wash Area No. 2 and the Building 837/Former Building 847 area. Additionally, after monitoring well installation, groundwater samples will be collected and analyzed for VOCs with select monitoring wells also sampled and analyzed for long-term monitoring parameters.

Below is a brief discussion of planned RI Addendum field activities at each of these OU 1 areas. WP Section 6.0/FSP Section 5.0 provides a complete discussion and sampling rationale for each area. FSP Section 6.0 provides descriptions for sample collection method(s). FSP Tables 5-2 through 5-6 presents the samples to be collected at each area and FSP Figures 5-1 through 5-8 illustrate the sample locations.

Jet Engine Repair Hangar/Aircraft Wash Area No. 2 (FSP Section 5.1/WP Section 6.1.3)

Sufficient surface and subsurface soil, sediment, surface water, and groundwater data are present to characterize this area. However, data gaps exist and bedrock core will be collected to determine the physical characteristics of the bedrock. The borehole will be completed as a bedrock monitoring well. The monitoring well will be sampled for VOCs and also possibly sampled for natural attenuation indicator parameters. FSP Table 5-2 presents the samples planned for collection and FSP Figure 5-1 illustrates the Jet Engine Repair Hangar/Aircraft Wash Area No. 2 areas.

Sewage Treatment Plant and Landfill Area (FSP Section 5.2/WP Section 6.2.3)

Surface and subsurface soil samples will be collected during a direct-push investigation from selected areas within the Sewage Treatment Plant and Landfill area. Surface soil samples will not be collected within the area of the concrete plant as this area has been extensively disturbed and covered with concrete. Subsurface soil samples will only be collected from areas assumed to be outside the limits of the Landfill. Surface soil samples will be collected and analyzed off-site for SVOCs and RCRA Metals. Subsurface soil samples will be collected and analyzed off-site for VOCs, SVOCs, and/or RCRA Metals, depending on the sample depth. Sediment and surface water samples will be collected from the drainage areas and ditches upgradient and downgradient of the area. These sediment and surface water samples will be analyzed off-site for VOCs, SVOCs, and RCRA Metals.

Groundwater samples will be collected using direct-push technology from locations assumed to be outside the limits of the Landfill and will be analyzed on-site for selected VOCs (tetrachloroethene [PCE],

trichloroethene [TCE], cis-1,2-dichloroethene [cis-1,2-DCE], and 1,1-dicloroethene [1,1-DCE]) using a field GC. One groundwater sample will be collected from the borehole for each 15 feet of saturated thickness of the aquifer. Confirmation samples will be collected at a rate of 10 percent and analyzed at an off-site laboratory for VOCs. FSP Table 5-3 presents the samples planned for collection and FSP Figure 5-2 illustrates the Sewage Treatment Plant and Landfill area.

LOX Plant (FSP Section 5.3/WP Section 6.3.3)

Surface and subsurface soil samples will be collected from selected areas during a direct-push investigation. Surface and subsurface soil samples will be analyzed off-site for VOCs and/or SVOCs, depending on sample depth. Sediment and surface water samples will be collected from the drainage areas and ditches upgradient and downgradient of the area . These sediment and surface water samples will be analyzed off-site for VOCs and SVOCs.

Groundwater samples will be collected using direct-push technology from locations around the LOX Plant and LOX Plant culvert/subsurface drains. Groundwater samples will be analyzed on-site for select VOCs (PCE, TCE, cis-1,2-DCE, and 1,1-DCE). Confirmation samples will be collected at a rate of 10 percent and analyzed at an off-site laboratory for VOCs.

If groundwater and/or soil samples indicate that the LOX Plant area may be a source for the contaminants located in groundwater to the northeast of the area, then a bedrock core will be collected. The borehole will be completed as a bedrock monitoring well and will be sampled for VOCs and also possibly sampled for natural attenuation indicator parameters. FSP Table 5-4 presents the samples planned for collection and FSP Figure 5-4 illustrates the LOX area.

Building 837 and Former Building 847 Area (FSP Section 5.4/WP Section 6.4.3)

A down-hole geophysics/conductivity survey is planned north of Building 837 to determine the top of bedrock. The borehole will be completed as a bedrock monitoring well and will be sampled for VOCs and also possibly sampled for natural attenuation indicator parameters.

A direct-push boring will be conducted northeast of Monitoring Well 99M24. Based upon the geologic characterization and the results of the on-site analysis of the groundwater samples, two additional monitoring wells, one shallow and one deep, will be installed in a cluster with 99M24. These monitoring wells and the bedrock monitoring well will be sampled and analyzed off-site for VOCs and may be selected to be analyzed for NA indicator parameters.

Soil samples will be collected from the area surrounding the Tullis Building. Kansas State University (KSU) is planning to place a day care facility within this building. The surface soil samples will be used to determine if risk is present within this area. Surface soil samples will be analyzed off-site for SVOCs and RCRA Metals. Subsurface soil samples will be collected and analyzed off-site for VOCs. FSP Table 5-5 presents the samples planned for collection and FSP Figure 5-5 illustrates the Building 837/Former Building 847 area.

Groundwater

Groundwater samples will be collected using direct-push technology from plume locations shown on FSP Figures 5-2 through 5-9 and will be analyzed on-site by field GC for selected VOCs (TCE, PCE, cis-1,2-DCE, 1,1-DCE) at Plume Areas A, B, C, D, and E. Additionally, to determine the extent of carbon tetrachloride contamination, one area in Plume C will also include on-site GC analysis of carbon tetrachloride, in addition to the previously mentioned VOC analytes. The results of the groundwater samples will be used to determine the vertical and horizontal extent of the groundwater contamination. FSP Tables 5-2 through 5-6 and FSP Figures 5-1 through 5-8 present the sampling requirements and locations for these groundwater plume areas.

Following the direct-push investigation, new monitoring wells in the above-mentioned areas may be installed after CENWK-approval to assist in the determination of groundwater contamination. VOCs will be collected from monitoring wells and analyzed off-site at AML for VOCs. Long-term monitoring parameters will also be collected and analyzed off-site at AML for 20 percent of these monitoring wells. Locations for the long-term monitoring well sampling will be determined once the configuration of the monitoring well network is known. The long-term monitoring parameters include VOCs and natural attenuation indicator parameters (MEE, iron, anions, sulfide, and TOC).

ANALYSIS TASKS:

Analysis tasks are noted above for each OU 1 area with planned field activities. FSP Tables 5-2 through 5-6 present the planned sampling locations and planned samples to be collected at each area.

In general, surface soil samples and subsurface soil samples will be collected and analyzed off-site by AML for VOCs, SVOCs, and/or RCRA Metals. Sediment and surface water from drainage ditches will be analyzed off-site by AML for VOCs, SVOCs, and/or RCRA Metals. Groundwater samples collected from direct-push activities will be analyzed on-site using a field GC for VOCs. Confirmation samples for these groundwater samples will be collected and analyzed off-site for VOCs by AML for 10 percent of the field samples.

Upon installation of the monitoring wells, VOCs samples will be collected and analyzed off-site. Additionally, long-term monitoring parameters will be collected and analyzed off-site at AML for 20 percent of these monitoring wells.

The primary analytical laboratory for off-site analysis is AML. EPS will perform on-site VOC analysis using a field GC. QA split samples from field activities will be sent off-site to a QA lab (ECB Laboratory) at a frequency of 10 percent. The analytical methods are also presented on Worksheets #19 and #23.

QUALITY CONTROL TASKS:

QC samples will be collected as described in FSP Tables 5-2 through 5-6.

See also Worksheet #28 which details relevant lab and field QC samples.

SECONDARY DATA:

See Worksheet #13 and WP Section 6.0.

OTHER DATA:

Data accumulated from the conductivity study and geophysics study is not included in this QAPP. Details regarding these studies can be found in FSP Section 5.0.

DATA MANAGEMENT TASKS:

Field notebooks, COC forms, field forms, disks, tapes, and lab reports will be filed and stored at the BMcD offices located in Kansas City, Missouri. After the first six months following completion of the project, these files may be transferred to long-term storage facilities located in Kansas City, Missouri or transferred to CENWK. Analytical data will be submitted in hard copy and electronically in SEDD 2a format.

DOCUMENTATION AND RECORDS:

AML data package deliverable requirements are outlined on QAPP Worksheet #29.

Project Reporting requirements are discussed in WP Section 5.0, including:

Remedial Investigation Report Addendum
Feasibility Study Report
Baseline Risk Assessment

Field operation documentation requirements are outlined in FSP Section 7.0, including:

7.1 DQCRs
7.2 Field Logbook and/or Sample Field Sheets

7.3 Photographic Records
7.4 Sample Documentation
7.5 Field Analytical Records
7.6 Documentation Procedures/Data Management and Retention
7.7 Corrections to Documentation

ASSESSMENT / AUDIT TASKS:

The following project tasks will be conducted for QA/QC assessment:

- DQCRs will be prepared by the FSM and reviewed by the project team
- Confirmation samples for groundwater are being collected during direct-push activities at 10 percent frequency and analyzed by an off-site lab
- QA split samples are being collected at 10 percent frequency and submitted to a secondary laboratory for analysis as part of a Split Sample Audit
- One double-blind PE sample for VOCs in groundwater will be submitted to AML for VOC analysis. Results submitted by AML will be compared to the laboratory-certified results of the PE sample.

These tasks are described on QAPP Worksheets #31 and #32.

DATA REVIEW TASKS:

Data review Steps I, IIa, IIb, and III will be performed on the soil and water data. Step I, Verification, is described on QAPP Worksheet #34. Steps IIa and IIb, Validation, are described on QAPP Worksheets #35 and #36. Step III, Data Usability Assessment, is described on QAPP Worksheet #37.

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QAPP Worksheet #15
(UFP-QAPP Manual Section 2.8.1)
Reference Limits and Evaluation Table

QAPP Worksheet #15-1 VOCs by SW-846 8260B (Soil/Sediment)

Matrix: Soil/Sediment
Analytical Group: VOCs by SW-846 8260B
Concentration Level: Low

Analyte	CAS Number	Action Limit[1] (µg/kg)	Project PQL Goal (µg/kg)	Analytical Method[2] SW-846 8260B		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/kg)	PQLs (µg/kg)	MDLs (µg/kg)	PQLs (µg/kg)
Acetone	67-64-1	54,000,000	18,000,000	Note 5	Note 6	2.51	5
Benzene	71-43-2	1400	470	Note 5	Note 6	0.8	5
Bromobenzene	108-86-1	92,000	31,000	Note 5	Note 6	0.8	5
Bromochloromethane	74-97-5	Note 4	NA	Note 5	Note 6	0.7	5
Bromodichloromethane	75-27-4	18,000	600	Note 5	Note 6	0.2	5
Bromoform	75-25-2	220,000	73,000	Note 5	Note 6	0.9	5
Bromomethane	74-83-9	13000	4300	Note 5	Note 6	1.6	5
2-Butanone	78-93-3	110,000,000	37,000,000	Note 5	Note 6	1.6	5
n-Butylbenzene	104-51-8	240,000	80,000	Note 5	Note 6	0.6	5
Sec-Butylbenzene	135-9-88	220,000	73,000	Note 5	Note 6	0.9	5
tert-Butylbenzene	98-06-6	390,000	130,000	Note 5	Note 6	0.6	5
Carbon Disulfide	75-15-0	720,000	240,000	Note 5	Note 6	0.9	5
Carbon Tetrachloride	56-23-5	550	180	Note 5	Note 6	1.7	5
Chlorobenzene	108-90-7	530,000	180,000	Note 5	Note 6	0.7	5
Chloroethane	75-00-3	6500	2200	Note 5	Note 6	1.9	5

Analyte	CAS Number	Action Limit[1] (µg/kg)	Project PQL Goal (µg/kg)	Analytical Method[2] SW-846 8260B		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/kg)	PQLs (µg/kg)	MDLs (µg/kg)	PQLs (µg/kg)
Chloroform	67-66-3	470	160	Note 5	Note 6	0.5	5
Chloromethane	74-87-3	160,000	53,000	Note 5	Note 6	1.1	5
2-Chlorotoluene	95-49-8	560,000	190,000	Note 5	Note 6	0.7	5
4-Chlorotoluene	106-43-4	Note 4	NA	Note 5	Note 6	0.5	5
1,2-Dibromo-3-chloropropane	96-12-8	2000	670	Note 5	Note 6	1.2	5
Dibromochloromethane	124-48-1	26,000	870	Note 5	Note 6	0.2	5
1,2-Dibromoethane	106-93-4	73	24	Note 5	Note 6	2.6	5
Dibromomethane	74-95-3	230,000	77,000	Note 5	Note 6	0.6	5
1,2-Dichlorobenzene	95-50-1	600,000	200,000	Note 5	Note 6	0.7	5
1,3-Dichlorobenzene	541-73-1	600,000	200,000	Note 5	Note 6	0.6	5
1,4-Dichlorobenzene	106-46-7	7900	2600	Note 5	Note 6	0.6	5
Dichlorodifluoromethane	75-71-8	310,000	100,000	Note 5	Note 6	1.3	5
1,1-Dichloroethane	75-34-3	1,700,000	570,000	Note 5	Note 6	0.4	5
1,2-Dichloroethane	107-06-2	600	200	Note 5	Note 6	0.6	5
1,1-Dichloroethene	75-35-4	4,100,000	140,000	Note 5	Note 6	0.6	5
cis-1,2-Dichloroethene	156-59-2	150,000	50,000	Note 5	Note 6	0.7	5
trans-1,2-Dichloroethene	156-60-5	230,000	77,000	Note 5	Note 6	0.6	5
1,2-Dichloropropane	78-87-5	740	250	Note 5	Note 6	0.7	5
1,3-Dichloropropane	142-28-9	360,000	120,000	Note 5	Note 6	0.4	5
2,2-Dichloropropane	594-20-7	Note 4	NA	Note 5	Note 6	0.4	5
1,1-Dichloropropene	563-58-6	Note 4	NA	Note 5	Note 6	0.3	5
cis-1,3-Dichloropropene	10061-01-5	Note 4	NA	Note 5	Note 6	0.6	5

Analyte	CAS Number	Action Limit[1] (µg/kg)	Project PQL Goal (µg/kg)	Analytical Method[2] SW-846 8260B		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/kg)	PQLs (µg/kg)	MDLs (µg/kg)	PQLs (µg/kg)
trans-1,3-Dichloropropene	10061-02-6	Note 4	NA	Note 5	Note 6	0.4	5
Ethylbenzene	100-41-4	400,000	130,000	Note 5	Note 6	0.6	5
Hexachlorobutadiene	87-68-3	22,000	7300	Note 5	Note 6	1.2	5
2-Hexanone	591-78-6	Note 4	NA	Note 5	Note 6	0.7	5
Isopropylbenzene	98-82-8	2,000,000	670,000	Note 5	Note 6	0.4	5
4-Isopropyltoluene	99-87-6	Note 4	NA	Note 5	Note 6	0.6	5
Methylene Chloride	75-09-2	21,000	7000	Note 5	Note 6	0.8	5
4-methyl-2-pentanone	108-10-1	47,000,000	16,000,000	Note 5	Note 6	0.9	5
Methyl-tert-butyl-ether	1634-04-4	70,000	23,000	Note 5	Note 6	0.35	5
Naphthalene	91-20-3	190,000	63,000	Note 5	Note 6	0.8	5
n-Propylbenzene	103-65-1	240,000	80,000	Note 5	Note 6	0.6	5
Styrene	100-42-5	1,700,000	570,000	Note 5	Note 6	0.5	5
1,1,1,2-Tetrachloroethane	630-20-6	7300	2400	Note 5	Note 6	0.7	5
1,1,2,2-Tetrachloroethane	79-34-5	930	310	Note 5	Note 6	1.2	5
Tetrachloroethene	127-18-4	1300	430	Note 5	Note 6	0.9	5
Toluene	108-88-3	520,000	170,000	Note 5	Note 6	0.4	5
1,2,3-Trichlorobenzene	87-61-6	Note 4	NA	Note 5	Note 6	1	5
1,2,4-Trichlorobenzene	120-82-1	220,000	73,000	Note 5	Note 6	0.9	5
1,1,1-Trichloroethane	71-55-6	1,200,000	400,000	Note 5	Note 6	1.2	5
1,1,2-Trichloroethane	79-00-5	1600	530	Note 5	Note 6	0.7	5
Trichloroethene	79-01-6	110	37	Note 5	Note 6	0.7	5
Trichlorofluoromethane	75-69-4	2,000,000	6,700,000	Note 5	Note 6	1.3	5

Analyte	CAS Number	Action Limit[1] (µg/kg)	Project PQL Goal (µg/kg)	Analytical Method[2] SW-846 8260B		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/kg)	PQLs (µg/kg)	MDLs (µg/kg)	PQLs (µg/kg)
1,2,3-Trichloropropane	96-18-4	760	250	Note 5	Note 6	0.9	5
1,2,4-Trimethylbenzene	95-63-6	170,000	57,000	Note 5	Note 6	0.7	5
1,3,5-Trimethylbenzene	108-67-8	70,000	23,000	Note 5	Note 6	0.8	5
Vinyl Chloride	75-01-4	750	250	Note 5	Note 6	1.1	5
m-Xylene and p-Xylene	m: 108-38-3 p: 106-42-3	420,000 t	140,000 t	Note 5	Note 6	1.5	5
o-Xylene	95-47-6	420,000 t	140,000 t	Note 5	Note 6	0.6	5

[1]ALs for soil and sediment matrices are USEPA PRGs and ALs for water matrices are MCLs. (See Worksheet #12)

[2]Analytical MDLs and PQLs are those documented in validated methods.

[3]Achievable MDLs and PQLs are limits that an individual laboratory can achieve when performing a specific analytical method. AML's achievable limits were determined based upon procedures presented in their SOP.

[4]There is no AL available for this parameter.

[5]MDLs should be equivalent to or below criteria given in SW-846 8260B.

[6]PQLs should be equivalent to or below criteria given in SW-846 8260B.

[(t)]Value represents AL and Project PQL Goal for total xylenes.

NA – Limit is not available. An AL is not established, hence the project PQL goal could not be determined.

See Worksheet #12, pages 12-33 to 12-34 for a discussion of sensitivity and PQLs. Acceptable results are obtained when the laboratory's PQL is less than the corresponding AL.

QAPP Worksheet #15-2 VOCs by SW-846 8260B (Water)

Matrix: Water
Analytical Group: VOCs by SW-846 8260B
Concentration Level: Low

Analyte	CAS Number	Action Limit[1] (µg/L)	Project PQL Goal (µg/L)	Analytical Method[2] SW-846 8260B		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/L)	PQLs (µg/L)	MDLs (µg/L)	PQLs (µg/L)
Acetone	67-64-1	Note 4	NA	Note 5	Note 6	0.6	1
Benzene	71-43-2	5	1.7	Note 5	Note 6	0.1	1
Bromobenzene	108-86-1	Note 4	NA	Note 5	Note 6	0.2	1
Bromochloromethane	74-97-5	Note 4	NA	Note 5	Note 6	0.2	1
Bromodichloromethane	75-27-4	80	27	Note 5	Note 6	0.1	1
Bromoform	75-25-2	80	27	Note 5	Note 6	0.2	1
Bromomethane	74-83-9	Note 4	NA	Note 5	Note 6	0.2	1
2-Butanone	78-93-3	Note 4	NA	Note 5	Note 6	0.5	1
n-Butylbenzene	104-51-8	Note 4	NA	Note 5	Note 6	0.1	1
Sec-Butylbenzene	135-9-88	Note 4	NA	Note 5	Note 6	0.1	1
tert-Butylbenzene	98-06-6	Note 4	NA	Note 5	Note 6	0.2	1
Carbon Disulfide	75-15-0	Note 4	NA	Note 5	Note 6	0.2	1
Carbon Tetrachloride	56-23-5	5	1.7	Note 5	Note 6	0.1	1
Chlorobenzene	108-90-7	100	33	Note 5	Note 6	0.2	1
Chloroethane	75-00-3	Note 4	NA	Note 5	Note 6	0.2	1
Chloroform	67-66-3	80	27	Note 5	Note 6	0.2	1
Chloromethane	74-87-3	Note 4	NA	Note 5	Note 6	0.2	1
2-Chlorotoluene	95-49-8	Note 4	NA	Note 5	Note 6	0.1	1

Analyte	CAS Number	Action Limit[1] (µg/L)	Project PQL Goal (µg/L)	Analytical Method[2] SW-846 8260B		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/L)	PQLs (µg/L)	MDLs (µg/L)	PQLs (µg/L)
4-Chlorotoluene	106-43-4	Note 4	NA	Note 5	Note 6	0.1	1
1,2-Dibromo-3-chloropropane	96-12-8	5	1.7	Note 5	Note 6	0.1	1
Dibromochloromethane	124-48-1	80	27	Note 5	Note 6	0.1	1
1,2-Dibromoethane	106-93-4	5	1.7	Note 5	Note 6	0.1	1
Dibromomethane	74-95-3	Note 4	NA	Note 5	Note 6	0.1	1
1,2-Dichlorobenzene	95-50-1	600	200	Note 5	Note 6	0.1	1
1,3-Dichlorobenzene	541-73-1	Note 4	NA	Note 5	Note 6	0.2	1
1,4-Dichlorobenzene	106-46-7	75	25	Note 5	Note 6	0.2	1
Dichlorodifluoromethane	75-71-8	Note 4	NA	Note 5	Note 6	0.5	1
1,1-Dichloroethane	75-34-3	Note 4	NA	Note 5	Note 6	0.2	1
1,2-Dichloroethane	107-06-2	5	1.7	Note 5	Note 6	0.2	1
1,1-Dichloroethene	75-35-4	7	2.3	Note 5	Note 6	0.2	1
cis-1,2-Dichloroethene	156-59-2	70	23	Note 5	Note 6	0.2	1
trans-1,2-Dichloroethene	156-60-5	100	33	Note 5	Note 6	0.2	1
1,2-Dichloropropane	78-87-5	5	1.7	Note 5	Note 6	0.2	1
1,3-Dichloropropane	142-28-9	Note 4	NA	Note 5	Note 6	0.1	1
2,2-Dichloropropane	594-20-7	Note 4	NA	Note 5	Note 6	0.1	1
1,1-Dichloropropene	563-58-6	Note 4	NA	Note 5	Note 6	0.1	1
cis-1,3-Dichloropropene	10061-01-5	Note 4	NA	Note 5	Note 6	0.1	1
trans-1,3-Dichloropropene	10061-02-6	Note 4	NA	Note 5	Note 6	0.1	1
Ethylbenzene	100-41-4	700	230	Note 5	Note 6	0.1	1
Hexachlorobutadiene	87-68-3	Note 4	NA	Note 5	Note 6	0.2	1

Analyte	CAS Number	Action Limit[1] (µg/L)	Project PQL Goal (µg/L)	Analytical Method[2] SW-846 8260B		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/L)	PQLs (µg/L)	MDLs (µg/L)	PQLs (µg/L)
2-Hexanone	591-78-6	Note 4	NA	Note 5	Note 6	0.2	1
Isopropylbenzene	98-82-8	Note 4	NA	Note 5	Note 6	0.1	1
4-Isopropyltoluene	99-87-6	Note 4	NA	Note 5	Note 6	0.1	1
Methylene Chloride	75-09-2	5	1.7	Note 5	Note 6	0.4	1
4-methyl-2-pentanone	108-10-1	Note 4	NA	Note 5	Note 6	0.1	1
Methyl-tert-butyl-ether	1634-04-4	Note 4	NA	Note 5	Note 6	0.1	1
Naphthalene	91-20-3	Note 4	NA	Note 5	Note 6	0.1	1
n-Propylbenzene	103-65-1	Note 4	NA	Note 5	Note 6	0.1	1
Styrene	100-42-5	100	33	Note 5	Note 6	0.1	1
1,1,1,2-Tetrachloroethane	630-20-6	Note 4	NA	Note 5	Note 6	0.2	1
1,1,2,2-Tetrachloroethane	79-34-5	Note 4	NA	Note 5	Note 6	0.1	1
Tetrachloroethene	127-18-4	5	1.7	Note 5	Note 6	0.1	1
Toluene	108-88-3	1000	330	Note 5	Note 6	0.1	1
1,2,3-Trichlorobenzene	87-61-6	Note 4	NA	Note 5	Note 6	0.1	1
1,2,4-Trichlorobenzene	120-82-1	70	23	Note 5	Note 6	0.1	1
1,1,1-Trichloroethane	71-55-6	200	67	Note 5	Note 6	0.2	1
1,1,2-Trichloroethane	79-00-5	5	1.7	Note 5	Note 6	0.1	1
Trichloroethene	79-01-6	5	1.7	Note 5	Note 6	0.2	1
Trichlorofluoromethane	75-69-4	Note 4	NA	Note 5	Note 6	0.1	1
1,2,3-Trichloropropane	96-18-4	Note 4	NA	Note 5	Note 6	0.1	1
1,2,4-Trimethylbenzene	95-63-6	Note 4	NA	Note 5	Note 6	0.1	1
1,3,5-Trimethylbenzene	108-67-8	Note 4	NA	Note 5	Note 6	0.1	1

Analyte	CAS Number	Action Limit[1] (µg/L)	Project PQL Goal (µg/L)	Analytical Method[2] SW-846 8260B		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/L)	PQLs (µg/L)	MDLs (µg/L)	PQLs (µg/L)
Vinyl Chloride	75-01-4	2	0.67	Note 5	Note 6	0.2	1
m-Xylene and p-Xylene	m: 108-38-3 p: 106-42-3	10,000 t	3300	Note 5	Note 6	0.2	1
o-Xylene	95-47-6	10,000 t	3300	Note 5	Note 6	0.1	1

[1]ALs for soil and sediment matrices are USEPA PRGs and ALs for water matrices are MCLs. (See Worksheet #12). Exceptions were made for 1,2-dibromo-3-chloropropane and 1,2-dibromoethane, which are not compounds of interest for the Site. Using the selected analytical techniques, it is not possible to achieve PQLs less than MCLs. Therefore, ALs of 5 µg/L were selected for these compounds. An AL of 5 µg/L is readily achievable using SW-846 8260B and is adequate for purposes of the RI Addendum.

[2]Analytical MDLs and PQLs are those documented in validated methods.

[3]Achievable MDLs and PQLs are limits that an individual laboratory can achieve when performing a specific analytical method. AML's achievable limits were determined based upon procedures presented in their SOP.

[4]There is no AL available for this parameter.

[5]MDLs should be equivalent to or below criteria given in SW-846 8260B.

[6]PQLs should be equivalent to or below criteria given in SW-846 8260B.

[(t)]Value represents AL and Project PQL Goal for total xylenes.

NA – Limit is not available. An AL is not established, hence the project PQL goal could not be determined.

See Worksheet #12, pages 12-33 to 12-34 for a discussion of sensitivity and PQLs. Acceptable results are obtained when the laboratory's PQL is less than the corresponding AL.

QAPP Worksheet #15-3 SVOCs by SW-846 8270D (Soil/Sediment)

Matrix: Soil/Sediment
Analytical Group: SVOCs by SW-846 8270D
Concentration Level: Low

Analyte	CAS Number	Action Limit[1] (µg/kg)	Project PQL Goal (µg/kg)	Analytical Method[2] SW-846 8270D		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/kg)	PQLs (µg/kg)	MDLs (µg/kg)	PQLs (µg/kg)
Acenaphthene	83-32-9	29000000	9700000	Note 5	Note 6	6	167
Acenaphthylene	83-32-9	29000000	9700000	Note 5	Note 6	4	167
Anthracene	120-12-7	100000000	33000000	Note 5	Note 6	7.2	167
Benzo(a)anthracene	56-55-3	2100	700	Note 5	Note 6	21.9	167
Benzo(b)fluoranthene	205-99-2	2100	700	Note 5	Note 6	55.5	167
Benzo(ghi)perylene	191-24-2	Note 4	NA	Note 5	Note 6	81.2	167
Benzo(k)fluoranthene	207-08-9	21000	7000	Note 5	Note 6	101	167
Benzoic Acid	65-85-0	100000000	10000000	Note 5	Note 6	81.2	167
Benzo(a)pyrene	50-32-8	2100	700	Note 5	Note 6	21.9	167
Benzyl Alcohol	100-51-6	100000000	33000000	Note 5	Note 6	81.2	167
bis(2-chloroethoxy)methane	111-91-1	Note 4	NA	Note 5	Note 6	7	167
bis(2-Chloroethyl)ether	111-44-4	580	190	Note 5	Note 6	8.5	167
bis(2-chloroisopropyl)ether	108-60-1	7400	2500	Note 5	Note 6	14.5	167
bis(2-ethylhexyl)phthalate	117-81-7	120000	40000	Note 5	Note 6	30.8	167
4-Bromophenyl-phenylether	101-55-3	Note 4	NA	Note 5	Note 6	11.9	167
Butylbenzylphthalate	85-68-7	100000000	33000000	Note 5	Note 6	13.5	167
Carbazole	86-74-8	86000	29000	Note 5	Note 6	33.3	167
4-Chloroaniline	106-47-8	2500000	250000	Note 5	Note 6	28.9	167

Analyte	CAS Number	Action Limit[1] (µg/kg)	Project PQL Goal (µg/kg)	Analytical Method[2] SW-846 8270D		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/kg)	PQLs (µg/kg)	MDLs (µg/kg)	PQLs (µg/kg)
4-Chloro-3-methylphenol	59-50-7	Note 4	NA	Note 5	Note 6	8.2	167
2-Chloronaphthalene	91-58-7	23000000	7700000	Note 5	Note 6	5.3	167
2-Chlorophenol	95-57-8	240000	80000	Note 5	Note 6	4.1	167
4-Chlorophenyl-phenyl ether	7005-72-3	Note 4	NA	Note 5	Note 6	25.5	167
Chrysene	218-01-9	210000	70000	Note 5	Note 6	13.2	167
Dibenz(a,h)anthracene	53-70-3	2100	700	Note 5	Note 6	75.3	167
Dibenzofuran	132-64-9	1600000	530000	Note 5	Note 6	5.6	167
1,2-Dichlorobenzene	95-50-1	600000	200000	Note 5	Note 6	7.7	167
1,3-Dichlorobenzene	541-73-1	600000	200000	Note 5	Note 6	12.1	167
1,4-Dichlorobenzene	106-46-7	7900	2600	Note 5	Note 6	6.5	167
3,3'-Dichlorobenzidine	91-94-1	3800	380	Note 5	Note 6	33	167
2,4-Dichlorophenol	120-83-2	1800000	600000	Note 5	Note 6	11.9	167
Diethylphthalate	84-66-2	100000000	33000000	Note 5	Note 6	13.5	167
2,4-Dimethylphenol	105-67-9	12000000	4000000	Note 5	Note 6	8.8	167
Dimethylphthalate	131-11-3	100000000	33000000	Note 5	Note 6	10.2	167
Di-n-butylphthalate	84-74-2	62000000	21000000	Note 5	Note 6	7.2	167
2,4-Dinitrophenol	51-28-5	1200000	400000	Note 5	Note 6	53.1	167
2,4-Dinitrotoluene	121-14-2	1200000	400000	Note 5	Note 6	11.7	167
2,6-Dinitrotoluene	606-20-2	6200000	2100000	Note 5	Note 6	8	167
Di-n-octylphthalate	117-84-0	25000000	8300000	Note 5	Note 6	62.7	167
Flouranthene	206-44-0	22000000	7300000	Note 5	Note 6	7.2	167
Fluorene	86-73-7	26000000	8700000	Note 5	Note 6	14.1	167

Analyte	CAS Number	Action Limit[1] (µg/kg)	Project PQL Goal (µg/kg)	Analytical Method[2] SW-846 8270D		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/kg)	PQLs (µg/kg)	MDLs (µg/kg)	PQLs (µg/kg)
Hexachlorobenzene	118-74-1	1100	370	Note 5	Note 6	20.7	167
Hexachlorobutadiene	87-68-3	22000	7300	Note 5	Note 6	31.6	167
Hexachlorocyclopentadiene	77-47-4	3700000	1200000	Note 5	Note 6	10.6	167
Hexachloroethane	67-72-1	120000	40000	Note 5	Note 6	10.2	167
Indeno(1,2,3-cd)pyrene	193-39-5	2100	700	Note 5	Note 6	108	167
Isophorone	78-59-1	510000	170000	Note 5	Note 6	4	167
2-Methyl-4,6-dinitrophenol	534-52-1	62000	21000	Note 5	Note 6	33.5	167
2-Methylnaphthalene	91-57-6	Note 4	NA	Note 5	Note 6	87.4	167
¾-Methylphenol (m/p-Cresol)	m: 108-39-4 p: 106-44-5	m: 31000 p: 3100	m: 10000 p: 1000	Note 5	Note 6	23.9	167
2-Methylphenol (o-Cresol)	95-48-7	Note 4	NA	Note 5	Note 6	5	167
Naphthanlene	91-20-3	31000000	10000000	Note 5	Note 6	7.9	167
2-Nitroaniline	88-74-4	190000	63000	Note 5	Note 6	14.4	167
3-Nitroaniline	99-09-2	1800000	600000	Note 5	Note 6	11.3	167
4-Nitroaniline	100-01-6	82000	27000	Note 5	Note 6	118	167
Nitrobenzene	98-95-3	82000	27000	Note 5	Note 6	10.9	167
2-Nitrophenol	88-75-5	100000	33000	Note 5	Note 6	11.8	167
4-Nitrophenol	100-02-7	Note 4	NA	Note 5	Note 6	27	167
N-Nitrosodimethylamine	62-75-9	Note 4	NA	Note 5	Note 6	10	167
N-Nitroso-di-n-propylamine	621-64-7	2500	830	Note 5	Note 6	11.9	167
N-Nitrosodiphenylamine	86-30-6	350000	116000	Note 5	Note 6	4.7	167
Pentachlorophenol	87-86-5	350000	120000	Note 5	Note 6	29.9	167

Analyte	CAS Number	Action Limit[1] (µg/kg)	Project PQL Goal (µg/kg)	Analytical Method[2] SW-846 8270D		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/kg)	PQLs (µg/kg)	MDLs (µg/kg)	PQLs (µg/kg)
Phenanthrene	85-01-8	9000	3000	Note 5	Note 6	8.1	167
Phenol	108-95-2	Note 4	NA	Note 5	Note 6	4	167
Pyrene	129-00-0	100000000	33000000	Note 5	Note 6	13.3	167
1,2,4-Trichlorobenzene	120-82-1	29000000	9700000	Note 5	Note 6	8.2	167
2,4,5-Trichlorophenol	95-95-4	220000	73000	Note 5	Note 6	28.6	167
2,4,6-Trichlorophenol	88-06-2	62000000	21000000	Note 5	Note 6	22.5	167

[1]ALs for soil and sediment matrices are USEPA PRGs and ALs for water matrices are MCLs. (See Worksheet #12). Exceptions were made for benzo(a)pyrene, dibenz(a,h)anthracene, and n-nitroso-di-n-propylamine, which are not the primary constituents of interest for the Site. Using the selected analytical technique, it is not possible to achieve PQLs less than 1/3 of the USEPA Region 9 PRG (i.e., PQL goal). Therefore, ALs that were 10 times the PRG were selected for these compounds. These ALs and subsequent PQL goals are readily achievable using SW-846 8270D and are adequate for purposes of the RI Addendum.

[2]Analytical MDLs and PQLs are those documented in validated methods.

[3]Achievable MDLs and PQLs are limits that an individual laboratory can achieve when performing a specific analytical method. AML's achievable limits were determined based upon procedures presented in their SOP.

[4]There is no AL available for this parameter.

[5]MDLs should be equivalent to or below criteria given in SW-846 8270D.

[6]PQLs should be equivalent to or below criteria given in SW-846 8270D.

NA – Limit is not available. An AL is not established, hence the project PQL goal could not be determined.

See Worksheet #12, pages 12-33 to 12-34 for a discussion of sensitivity and PQLs. Acceptable results are obtained when the laboratory's PQL is less than the corresponding AL.

QAPP Worksheet #15-4 SVOCs by SW-846 8270D (Water)

Matrix: Water
Analytical Group: SVOCs by SW-846 8270D
Concentration Level: Low

Analyte	CAS Number	Action Limit[1] (µg/L)	Project PQL Goal (µg/L)	Analytical Method[2] SW-846 8270D		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/L)	PQLs (µg/L)	MDLs (µg/L)	PQLs (µg/L)
Acenaphthene	83-32-9	Note 4	NA	Note 5	Note 6	0.2	5
Acenaphthylene	83-32-9	Note 4	NA	Note 5	Note 6	0.1	5
Anthracene	120-12-7	Note 4	NA	Note 5	Note 6	0.2	5
Benzo(a)anthracene	56-55-3	Note 4	NA	Note 5	Note 6	0.1	5
Benzo(b)fluoranthene	205-99-2	Note 4	NA	Note 5	Note 6	0.09	5
Benzo(ghi)perylene	191-24-2	Note 4	NA	Note 5	Note 6	0.44	5
Benzo(k)fluoranthene	207-08-9	Note 4	NA	Note 5	Note 6	0.9	5
Benzoic Acid	65-85-0	Note 4	NA	Note 5	Note 6	2.4	5
Benzo(a)pyrene	50-32-8	20	6.7	Note 5	Note 6	0.1	5
Benzyl Alcohol	100-51-6	Note 4	NA	Note 5	Note 6	0.5	5
bis(2-chloroethoxy)methane	111-91-1	Note 4	NA	Note 5	Note 6	0.2	5
bis(2-Chloroethyl)ether	111-44-4	Note 4	NA	Note 5	Note 6	0.1	5
bis(2-chloroisopropyl)ether	108-60-1	Note 4	NA	Note 5	Note 6	0.4	5
bis(2-ethylhexyl)phthalate	117-81-7	60	20	Note 5	Note 6	0.6	5
4-Bromophenyl-phenylether	101-55-3	Note 4	NA	Note 5	Note 6	0.4	5
Butylbenzylphthalate	85-68-7	Note 4	NA	Note 5	Note 6	0.4	5
Carbazole	86-74-8	Note 4	NA	Note 5	Note 6	0.3	5
4-Chloroaniline	106-47-8	Note 4	NA	Note 5	Note 6	0.9	5

Analyte	CAS Number	Action Limit[1] (µg/L)	Project PQL Goal (µg/L)	Analytical Method[2] SW-846 8270D		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/L)	PQLs (µg/L)	MDLs (µg/L)	PQLs (µg/L)
4-Chloro-3-methylphenol	59-50-7	Note 4	NA	Note 5	Note 6	0.3	5
2-Chloronaphthalene	91-58-7	Note 4	NA	Note 5	Note 6	0.2	5
2-Chlorophenol	95-57-8	Note 4	NA	Note 5	Note 6	0.1	5
4-Chlorophenyl-phenyl ether	7005-72-3	Note 4	NA	Note 5	Note 6	0.5	5
Chrysene	218-01-9	Note 4	NA	Note 5	Note 6	0.4	5
Dibenz(a,h)anthracene	53-70-3	Note 4	NA	Note 5	Note 6	2.3	5
Dibenzofuran	132-64-9	Note 4	NA	Note 5	Note 6	0.2	5
1,2-Dichlorobenzene	95-50-1	600	200	Note 5	Note 6	0.2	5
1,3-Dichlorobenzene	541-73-1	Note 4	NA	Note 5	Note 6	0.4	5
1,4-Dichlorobenzene	106-46-7	75	25	Note 5	Note 6	0.2	5
3,3'-Dichlorobenzidine	91-94-1	Note 4	NA	Note 5	Note 6	1.5	5
2,4-Dichlorophenol	120-83-2	Note 4	NA	Note 5	Note 6	0.4	5
Diethylphthalate	84-66-2	Note 4	NA	Note 5	Note 6	0.4	5
2,4-Dimethylphenol	105-67-9	Note 4	NA	Note 5	Note 6	0.2	5
Dimethylphthalate	131-11-3	Note 4	NA	Note 5	Note 6	0.3	5
Di-n-butylphthalate	84-74-2	Note 4	NA	Note 5	Note 6	0.2	5
2,4-Dinitrophenol	51-28-5	Note 4	NA	Note 5	Note 6	1.6	5
2,4-Dinitrotoluene	121-14-2	Note 4	NA	Note 5	Note 6	0.4	5
2,6-Dinitrotoluene	606-20-2	Note 4	NA	Note 5	Note 6	0.2	5
Di-n-octylphthalate	117-84-0	Note 4	NA	Note 5	Note 6	1.9	5
Flouranthene	206-44-0	Note 4	NA	Note 5	Note 6	0.2	5
Fluorene	86-73-7	Note 4	NA	Note 5	Note 6	0.4	5

Analyte	CAS Number	Action Limit[1] (µg/L)	Project PQL Goal (µg/L)	Analytical Method[2] SW-846 8270D		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/L)	PQLs (µg/L)	MDLs (µg/L)	PQLs (µg/L)
Hexachlorobenzene	118-74-1	15	5	Note 5	Note 6	0.1	5
Hexachlorobutadiene	87-68-3	Note 4	NA	Note 5	Note 6	1.0	5
Hexachlorocyclopentadiene	77-47-4	Note 4	NA	Note 5	Note 6	0.3	5
Hexachloroethane	67-72-1	Note 4	NA	Note 5	Note 6	0.3	5
Indeno(1,2,3-cd)pyrene	193-39-5	Note 4	NA	Note 5	Note 6	1.6	5
Isophorone	78-59-1	Note 4	NA	Note 5	Note 6	0.1	5
2-Methyl-4,6-dinitrophenol	534-52-1	Note 4	NA	Note 5	Note 6	1.0	5
2-Methylnaphthalene	91-57-6	Note 4	NA	Note 5	Note 6	2.6	5
¾-Methylphenol (m/p-Cresol)	m: 108-39-4 p: 106-44-5	Note 4	NA	Note 5	Note 6	0.2	5
2-Methylphenol (o-Cresol)	95-48-7	Note 4	NA	Note 5	Note 6	0.3	5
Naphthanlene	91-20-3	Note 4	NA	Note 5	Note 6	0.2	5
2-Nitroaniline	88-74-4	Note 4	NA	Note 5	Note 6	0.4	5
3-Nitroaniline	99-09-2	Note 4	NA	Note 5	Note 6	0.3	5
4-Nitroaniline	100-01-6	Note 4	NA	Note 5	Note 6	3.5	5
Nitrobenzene	98-95-3	Note 4	NA	Note 5	Note 6	0.3	5
2-Nitrophenol	88-75-5	Note 4	NA	Note 5	Note 6	0.4	5
4-Nitrophenol	100-02-7	Note 4	NA	Note 5	Note 6	0.8	5
N-Nitrosodimethylamine	62-75-9	Note 4	NA	Note 5	Note 6	0.4	5
N-Nitroso-di-n-propylamine	621-64-7	Note 4	NA	Note 5	Note 6	0.4	5
N-Nitrosodiphenylamine	86-30-6	Note 4	NA	Note 5	Note 6	0.1	5
Pentachlorophenol	87-86-5	15	5	Note 5	Note 6	0.3	5
Phenanthrene	85-01-8	Note 4	NA	Note 5	Note 6	0.2	5

Analyte	CAS Number	Action Limit[1] (µg/L)	Project PQL Goal (µg/L)	Analytical Method[2] SW-846 8270D		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/L)	PQLs (µg/L)	MDLs (µg/L)	PQLs (µg/L)
Phenol	108-95-2	Note 4	NA	Note 5	Note 6	0.1	5
Pyrene	129-00-0	Note 4	NA	Note 5	Note 6	0.4	5
1,2,4-Trichlorobenzene	120-82-1	70	23	Note 5	Note 6	0.3	5
2,4,5-Trichlorophenol	95-95-4	Note 4	NA	Note 5	Note 6	0.9	5
2,4,6-Trichlorophenol	88-06-2	Note 4	NA	Note 5	Note 6	0.7	5

[1]ALs for soil and sediment matrices are USEPA PRGs and ALs for water matrices are MCLs. (See Worksheet #12). Exceptions were made for benzo(a)pyrene, bis(2-ethylhexyl)phthalate, hexachlorobenzene, and pentachlorophenol, which are not the primary constituents of interest for the Site. Using the selected analytical technique, it is not possible to achieve PQLs less than the MCLs. Therefore, ALs that were 10 times the MCL were selected for benzo(a)pyrene and bis(2-ethylhexyl)phthalate. Additionally, ALs that were 15 times the MCL were selected for hexachlorobenzene and pentachlorophenol. These ALs and subsequent PQL goals are readily achievable using SW-846 8270D and are adequate for purposes of the RI Addendum.

[2]Analytical MDLs and PQLs are those documented in validated methods.

[3]Achievable MDLs and PQLs are limits that an individual laboratory can achieve when performing a specific analytical method. AML's achievable limits were determined based upon procedures presented in their SOP.

[4]There is no AL available for this parameter.

[5]MDLs should be equivalent to or below criteria given in SW-846 8270D.

[6]PQLs should be equivalent to or below criteria given in SW-846 8270D.

NA – Limit is not available. An AL is not established, hence the project PQL goal could not be determined.

See Worksheet #12, pages 12-33 to 12-34 for a discussion of sensitivity and PQLs. Acceptable results are obtained when the laboratory's PQL is less than the corresponding AL.

QAPP Worksheet #15-5 MEE by RSK-175 (Water)

Matrix: Water
Analytical Group: MEE by RSK-175
Concentration Level: Low

Analyte	CAS Number	Action Limit[1] (µg/L)	Project PQL Goal (µg/L)	Analytical Method[2] RSK-175		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/L)	PQLs (µg/L)	MDLs (µg/L)	PQLs (µg/L)
Methane	74-82-8	Note 4	NA	Note 5	Note 6	5	10
Ethane	74-84-0	Note 4	NA	Note 5	Note 6	10	20
Ethene	74-85-1	Note 4	NA	Note 5	Note 6	10	20

[1]ALs for soil and sediment matrices are USEPA PRGs and ALs for water matrices are MCLs. (See Worksheet #12)

[2]Analytical MDLs and PQLs are those documented in validated methods.

[3]Achievable MDLs and PQLs are limits that an individual laboratory can achieve when performing a specific analytical method. AML's achievable limits were determined based upon procedures presented in their SOP.

[4]There is no AL available for this parameter.

[5]MDLs should be equivalent to or below criteria given in RSK-175 or equivalent.

[6]PQLs should be equivalent to or below criteria given in RSK-175 or equivalent.

NA – Limit is not available. An AL is not established, hence the project PQL goal could not be determined.

See Worksheet #12, pages 12-33 to 12-34 for a discussion of sensitivity and PQLs. Acceptable results are obtained when the laboratory's PQL is less than the corresponding AL.

QAPP Worksheet #15-6 Metals by SW-846 Method 6020A (Soil/Sediment)

Matrix: Soil/Sediment
Analytical Group: Metals by SW-846 6020A
Concentration Level: Low

Analyte	CAS Number	Action Limit[1] (mg/kg)	Project PQL Goal (mg/kg)	Analytical Method[2] SW-846 6020A		AML's Achievable Laboratory Limits[3]	
				MDLs (mg/kg)	PQLs (mg/kg)	MDLs (mg/kg)	PQLs (mg/kg)
Arsenic	7440-38-2	1.6	0.53	Note 5	Note 6	0.04	0.08
Barium	7440-39-3	67000	22300	Note 5	Note 6	0.02	0.04
Cadmium	7440-43-9	450	150	Note 5	Note 6	0.02	0.04
Chromium	7440-47-3	450	150	Note 5	Note 6	0.1	0.2
Lead	7439-92-1	800	267	Note 5	Note 6	0.02	0.04
Selenium	7782-49-2	5100	1700	Note 5	Note 6	0.02	0.04
Silver	7440-22-4	5100	1700	Note 5	Note 6	0.02	0.04

[1]ALs for soil and sediment matrices are USEPA PRGs and ALs for water matrices are MCLs. (See Worksheet #12)

[2]Analytical MDLs and PQLs are those documented in validated methods.

[3]Achievable MDLs and PQLs are limits that an individual laboratory can achieve when performing a specific analytical method. AML's achievable limits were determined based upon procedures presented in their SOP.

[4]There is no AL available for this parameter.

[5]MDLs should be equivalent to or below criteria given in SW-846 6020A.

[6]PQLs should be equivalent to or below criteria given in SW-846 6020A.

See Worksheet #12, pages 12-33 to 12-34 for a discussion of sensitivity and PQLs. Acceptable results are obtained when the laboratory's PQL is less than the corresponding AL.

QAPP Worksheet #15-7 Mercury by SW-846 7471A (Soil/Sediment)

Matrix: Soil/Sediment
Analytical Group: Mercury by SW-846 7471A
Concentration Level: Low

Analyte	CAS Number	Action Limit[1] (µg/kg)	Project PQL Goal (µg/kg)	Analytical Method[2] SW-846 7471A		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/kg)	PQLs (µg/kg)	MDLs (µg/kg)	PQLs (µg/kg)
Mercury	7439-97-6	Note 4	NA	Note 5	Note 6	8	17

[1]ALs for soil and sediment matrices are USEPA PRGs and ALs for water matrices are MCLs. (See Worksheet #12)

[2]Analytical MDLs and PQLs are those documented in validated methods.

[3]Achievable MDLs and PQLs are limits that an individual laboratory can achieve when performing a specific analytical method. AML's achievable limits were determined based upon procedures presented in their SOP.

[4]There is no AL available for this parameter.

[5]MDLs should be equivalent to or below criteria given in SW-846 7471A or equivalent.

[6]PQLs should be equivalent to or below criteria given in SW-846 7471A or equivalent.

NA – Limit is not available. An AL is not established, hence the project PQL goal could not be determined.

See Worksheet #12, pages 12-33 to 12-34 for a discussion of sensitivity and PQLs. Acceptable results are obtained when the laboratory's PQL is less than the corresponding AL.

QAPP Worksheet #15-8 Metals by SW-846 6020A (Water)

Matrix: Water
Analytical Group: Metals by SW-846 6020A
Concentration Level: Low

Analyte	CAS Number	Action Limit[1] (mg/L)	Project PQL Goal (mg/L)	Analytical Method[2] SW-846 6020A		AML's Achievable Laboratory Limits[3]	
				MDLs[4] (mg/L)	PQLs[5] (mg/L)	MDLs (mg/L)	PQLs (mg/L)
Arsenic	7440-38-2	10	0.33	Note 6	Note 7	0.001	0.002
Barium	7440-39-3	2	0.67	Note 6	Note 7	0.0005	0.001
Cadmium	7440-43-9	5	1.7	Note 6	Note 7	0.0005	0.001
Chromium	7440-47-3	100	33	Note 6	Note 7	0.0025	0.005
Iron, Total	7439-89-6	0.30 (Note 4)	0.10	Note 6	Note 7	0.05	0.1
Lead	7439-92-1	0.15 (Note 5)	0.05	Note 6	Note 7	0.0005	0.001
Selenium	7782-49-2	50	16	Note 6	Note 7	0.001	0.002
Silver	7440-22-4	0.10 (Note 4)	0.03	Note 6	Note 7	0.0005	0.001

[1]ALs for soil and sediment matrices are USEPA PRGs and ALs for water matrices are MCLs. (See Worksheet #12)

[2]Analytical MDLs and PQLs are those documented in validated methods.

[3]Achievable MDLs and PQLs are limits that an individual laboratory can achieve when performing a specific analytical method. AML's achievable limits were determined based upon procedures presented in their SOP.

[4]Secondary MCL used for AL.

[5]AL used for lead is based on a treatment technique. AL for lead based on this treatment technique is as noted.

[6]MDLs should be equivalent to or below criteria given in SW-846 6020A.

[7]PQLs should be equivalent to or below criteria given in SW-846 6020A.

See Worksheet #12, pages 12-33 to 12-34 for a discussion of sensitivity and PQLs. Acceptable results are obtained when the laboratory's PQL is less than the corresponding AL.

QAPP Worksheet #15-9 Mercury by SW-846 7470A (Water)

Matrix: Water
Analytical Group: Mercury by SW-846 7470A
Concentration Level: Low

Analyte	CAS Number	Action Limit[1] (µg/L)	Project PQL Goal (µg/L)	Analytical Method[2] SW-846 7470A		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/L)	PQLs (µg/L)	MDLs (µg/L)	PQLs (µg/L)
Mercury	7439-97-6	2	0.67	Note 4	Note 5	0.1	0.2

[1]Analytical MDLs and PQLs are those documented in validated methods.

[2]Achievable MDLs and MQLs are limits that an individual laboratory can achieve when performing a specific analytical method. AML's achievable limits were determined based upon procedures presented in their SOP.

[3]MDLs should be equivalent to or below criteria given in SW-846 Method 7470A or equivalent.

[4]PQLs should be equivalent to or below criteria given in SW-846 Method 7470A or equivalent.

See Worksheet #12, pages 12-33 to 12-34 for a discussion of sensitivity and PQLs. Acceptable results are obtained when the laboratory's PQL is less than the corresponding AL.

QAPP Worksheet #15-10 Anions by EPA Method 300.0 (Water)

Matrix: Water
Analytical Group: Anions (Chloride, Nitrate, Nitrite, Sulfate) by EPA Method 300.0
Concentration Level: Low

Analyte	CAS Number	Action Limit[1] (mg/L)	Project PQL Goal (mg/L)	Analytical Method[2] EPA Method 300.0		AML's Achievable Laboratory Limits[3]	
				MDLs (mg/L)	PQLs (mg/L)	MDLs (mg/L)	PQLs (mg/L)
Chloride	16887-00-6	250 (Note 4)	83	Note 5	Note 6	0.1	0.5
Nitrate	14797-55-8	10	3.3	Note 5	Note 6	0.05	0.23
Nitrite	14797-65-0	1	0.33	Note 5	Note 6	0.08	0.38
Sulfate	14808-79-8	250 (Note 4)	83	Note 5	Note 6	0.2	1.00

[1]ALs for soil and sediment matrices are USEPA PRGs and ALs for water matrices are MCLs. (See Worksheet #12)

[2]Analytical MDLs and PQLs are those documented in validated methods.

[3]Achievable MDLs and PQLs are limits that an individual laboratory can achieve when performing a specific analytical method. AML's achievable limits were determined based upon procedures presented in their SOP.

[4]Secondary MCL used for AL.

[5]MDLs should be equivalent to or below criteria given in EPA Method 300.0.

[6]PQLs should be equivalent to or below criteria given in EPA Method 300.0.

See Worksheet #12, pages 12-33 to 12-34 for a discussion of sensitivity and PQLs. Acceptable results are obtained when the laboratory's PQL is less than the corresponding AL.

QAPP Worksheet #15-11 Sulfide by EPA Method 376.1 (Water)

Matrix: Water
Analytical Group: Sulfide by EPA Method 376.1
Concentration Level: Low

Analyte	CAS Number	Action Limit[1] (mg/L)	Project PQL Goal (mg/L)	Analytical Method[2] EPA Method 376.1		AML's Achievable Laboratory Limits[3]	
				MDLs (mg/L)	PQLs (mg/L)	MDLs (mg/L)	PQLs (mg/L)
Sulfide	18496-25-8	Note 4	NA	Note 5	Note 6	0.05	0.178

[1]ALs for soil and sediment matrices are USEPA PRGs and ALs for water matrices are MCLs. (See Worksheet #12)

[2]Analytical MDLs and PQLs are those documented in validated methods.

[3]Achievable MDLs and PQLs are limits that an individual laboratory can achieve when performing a specific analytical method. AML's achievable limits were determined based upon procedures presented in their SOP.

[4]There is no AL available for this parameter.

[5]MDLs should be equivalent to or below criteria given in EPA Method 376.1 or equivalent.

[6]PQLs should be equivalent to or below criteria given in EPA Method 376.1 or equivalent.

NA – Limit is not available. An AL is not established, hence the project PQL goal could not be determined.

See Worksheet #12, pages 12-33 to 12-34 for a discussion of sensitivity and PQLs. Acceptable results are obtained when the laboratory's PQL is less than the corresponding AL.

QAPP Worksheet #15-12 TOC by SW-846 9060

Matrix: Water
Analytical Group: TOC by SW-846 9060
Concentration Level: Low

Analyte	CAS Number	Action Limit[1] (µg/L)	Project PQL Goal (µg/L)	Analytical Method[2] SW-846 9060		AML's Achievable Laboratory Limits[3]	
				MDLs (µg/L)	PQLs (µg/L)	MDLs (mg/L)	PQLs (mg/L)
TOC	7440-44-0	Note 4	NA	Note 5	Note 6	0.5	1.0

[1]ALs for soil and sediment matrices are USEPA PRGs and ALs for water matrices are MCLs. (See Worksheet #12)

[2]Analytical MDLs and PQLs are those documented in validated methods.

[3]Achievable MDLs and PQLs are limits that an individual laboratory can achieve when performing a specific analytical method. AML's achievable limits were determined based upon procedures presented in their SOP.

[4]There is no AL available for this parameter.

[5]MDLs should be equivalent to or below criteria given in SW-846 9060 or equivalent.

[6]PQLs should be equivalent to or below criteria given in SW-846 9060 or equivalent.

NA – Limit is not available. An AL is not established, hence the project PQL goal could not be determined.

See Worksheet #12, pages 12-33 to 12-34 for a discussion of sensitivity and PQLs. Acceptable results are obtained when the laboratory's PQL is less than the corresponding AL.

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QAPP Worksheet #16
(UFP-QAPP Manual Section 2.8.2)
Project Schedule / Timeline Table

Activities	Organization	Dates		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
RI	BMcD and MP	2nd Quarter 2006	1st Quarter 2008		
RI Addendum WP	BMcD and MP	2nd Quarter 2006	4th Quarter 2006	RI Addendum WP	4th Quarter 2006
RI Addendum Field Work	BMcD and MP	4th Quarter 2006	2nd Quarter 2007	Field Notes and Field Documents Lab Deliverables for samples (on-site and off-site analysis) QCSR	2nd Quarter 2007
RI Report Addendum	BMcD and MP	2nd Quarter 2007	1st Quarter 2008	RI Report Addendum	1st Quarter 2008
BLRA	BMcD and MP	2nd Quarter 2007	1st Quarter 2008	BLRA Addendum	1st Quarter 2008
Feasibility Study	BMcD and MP	1st Quarter 2008	3rd Quarter 2008	Feasibility Study Report	3rd Quarter 2008
Decision Document	BMcD and MP	2nd Quarter 2008	3rd Quarter 2009	Proposed Plan Decision Document	3rd Quarter 2009
Remedial Design	BMcD and MP	2nd Quarter 2009	2nd Quarter 2010	Remedial Design Work Plan	2nd Quarter 2010
Remedial Action	BMcD, MP, KDHE, CENWK	2nd Quarter 2010	3rd Quarter 2012	Remedial Design Report	3rd Quarter 2012
Groundwater Monitoring	BMcD and MP	3rd Quarter 2006	3rd Quarter 2012	Field Notes and Field Documents Lab Deliverables for samples (on-site and off-site analysis)	3rd Quarter 2012

Activities	Organization	Dates		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Interim Remedial Action	BMcD and MP	2nd Quarter 2006	3rd Quarter 2009	Focused Feasibility Study Proposed Plan Interim Decision Document Interim Remedial Design Work Plan Interim Remedial Design Report	3rd Quarter 2009
Note: This schedule will be routinely updated by BMcD and MP during the course of the project. A more detailed project schedule is maintained by the CENWK, MP, and BMcD Project Managers.					

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QAPP Worksheet #17
(UFP-QAPP Manual Section 3.1.1)
Sampling Design and Rationale

Describe and provide rationale for choosing the sampling approach:

The overall rationale for the RI is presented in WP Sections 4.0 and 6.0. The field investigation activities will be conducted in four areas at the Site: Jet Engine Repair Hangar/Aircraft Wash Area No. 2, Sewage Treatment Plant and Landfill area, LOX Plant, and Building 837/Former Building 847 area.

Jet Engine Repair Hangar/Aircraft Wash Area No. 2 (FSP Section 5.1/WP Section 6.1.3)

Sufficient surface and subsurface soil, sediment, surface water, and groundwater data are present to characterize this area. However, data gaps exist and bedrock core will be collected to determine the physical characteristics of the bedrock. The borehole will be completed as a bedrock monitoring well. The monitoring well will be sampled for VOCs and also possibly sampled for natural attenuation indicator parameters. FSP Table 5-2 presents the samples planned for collection and FSP Figure 5-1 illustrates the Jet Engine Repair Hangar/Aircraft Wash Area No. 2 areas.

Sewage Treatment Plant and Landfill Area (FSP Section 5.2/WP Section 6.2.3)

Surface and subsurface soil samples will be collected during a direct-push investigation from selected areas within the Sewage Treatment Plant and Landfill area. Surface soil samples will not be collected within the area of the concrete plant as this area has been extensively disturbed and covered with concrete. Subsurface soil samples will only be collected from areas assumed to be outside the limits of the Landfill. Surface soil samples will be collected and analyzed off-site for SVOCs and RCRA Metals. Subsurface soil samples will be collected and analyzed off-site for VOCs, SVOCs, and/or RCRA Metals, depending on the sample depth. Sediment and surface water samples will be collected from the drainage areas and ditches upgradient and downgradient of the area. These sediment and surface water samples will be analyzed off-site for VOCs, SVOCs, and RCRA Metals.

Groundwater samples will be collected using direct-push technology from locations assumed to be outside the limits of the Landfill and will be analyzed on-site for selected VOCs (tetrachloroethene [PCE], trichloroethene [TCE], cis-1,2-dichloroethene [cis-1,2-DCE], and 1,1-dicloroethene [1,1-DCE]) using a field GC. One groundwater sample will be collected from the borehole for each 15 feet of saturated thickness of the aquifer. Confirmation samples will be collected at a rate of 10 percent and analyzed at an off-site laboratory for VOCs. FSP Table 5-3 presents the samples planned for collection and FSP Figure 5-2 illustrates the Sewage Treatment Plant and Landfill area.

LOX Plant (FSP Section 5.3/WP Section 6.3.3)

Surface and subsurface soil samples will be collected from selected areas during a direct-push investigation. Surface and subsurface soil samples will be analyzed off-site for VOCs and/or SVOCs, depending on sample depth. Sediment and surface water samples will be collected from the drainage areas and ditches upgradient and downgradient of the area . These sediment and surface water samples will be analyzed off-site for VOCs and SVOCs.

Groundwater samples will be collected using direct-push technology from locations around the LOX Plant and LOX Plant culvert/subsurface drains. Groundwater samples will be analyzed on-site for select VOCs (PCE, TCE, cis-1,2-DCE, and 1,1-DCE). Confirmation samples will be collected at a rate of 10 percent and analyzed at an off-site laboratory for VOCs.

If groundwater and/or soil samples indicate that the LOX Plant area may be a source for the contaminants located in groundwater to the northeast of the area, then a bedrock core will be collected. The borehole will be completed as a bedrock monitoring well and will be analyzed off-site for VOCs and also possibly sampled for natural attenuation indicator parameters. FSP Table 5-4 presents the samples planned for collection and FSP Figure 5-4 illustrates the LOX area.

Building 837 and Former Building 847 Area (FSP Section 5.4/WP Section 6.4.3)

A down-hole geophysics/conductivity survey is planned north of Building 837 to determine the top of bedrock. The borehole will be completed as a bedrock monitoring well and will be sampled for VOCs and also possibly sampled for natural attenuation indicator parameters.

A direct-push boring will be conducted northeast of Monitoring Well 99M24. Based upon the geologic characterization and the results of the on-site analysis of the groundwater samples, two additional monitoring wells, one shallow and one deep, will be installed in a cluster with 99M24. These monitoring wells and the bedrock monitoring well will be sampled and analyzed off-site for VOCs and may be selected to be analyzed for NA indicator parameters.

Soil samples will be collected from the area surrounding the Tullis Building. Kansas State University (KSU) is planning to place a day care facility within this building. The surface soil samples will be used to determine if risk is present within this area. Surface soil samples will be analyzed off-site for SVOCs and RCRA Metals. Subsurface soil samples will be collected and analyzed off-site for VOCs. FSP Table 5-5 presents the samples planned for collection and FSP Figure 5-5 illustrates the Building 837/Former Building 847 area.

Groundwater

Groundwater samples will be collected using direct-push technology from plume locations shown on FSP Figures 5-2 through 5-9 and will be analyzed on-site by field GC for selected VOCs (TCE, PCE, cis-1,2-DCE, 1,1-DCE) at Plume Areas A, B, C, D, and E. Additionally, to determine the extent of carbon tetrachloride contamination, one area in Plume C will also include on-site GC analysis of carbon tetrachloride, in addition to the previously mentioned VOC analytes. The results of the groundwater samples will be used to determine the vertical and horizontal extent of the groundwater contamination. FSP Tables 5-2 through 5-6 and FSP Figures 5-1 through 5-8 present the sampling requirements and locations for these groundwater plume areas.

Following the direct-push investigation, new monitoring wells in the above-mentioned areas may be installed after CENWK-approval to assist in the determination of groundwater contamination. VOCs will be collected from monitoring wells and analyzed off-site at AML for VOCs. Long-term monitoring parameters will also be collected and analyzed off-site at AML for 20 percent of these monitoring wells. Locations for the long-term monitoring well sampling will be determined once the configuration of the monitoring well network is known. The long-term monitoring parameters include VOCs and natural attenuation indicator parameters (MEE, iron, anions, sulfide, and TOC).

Describe the sampling design rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations, the number of samples to be taken, and the sampling frequency:

See also QAPP Worksheet #18 for sampling methods and requirements and FSP Tables 5-2 through 5-6 for sample locations and analyses.

The number of samples to be collected will be determined based on the specific area and field investigation data. Soil, sediment, floor drain, surface water, and/or groundwater samples will be sampled and analyzed as presented in FSP Section 5.0 and associated FSP Tables 5-2 through 5-6. Planned sampling locations are presented on FSP Figures 5-1 through 5-8. All concentrations will be reported at a low concentration level (See Worksheet #15).

QC activities include the collection of field duplicate and QA split samples with a frequency of 10 percent. MS/MSD will be collected with a frequency of 5 percent to account for reduced holding times of particular analyses. Rinsate blanks will be collected with a frequency of 20 percent when non-dedicated equipment is used. Rinsate blanks will be collected for analysis at least once per week but no more than

once per day. Trip blank samples will be submitted with all shipments of off-site aqueous VOC and MEE samples. For all on-site VOC analysis using a field GC, confirmation groundwater samples will be sent to AML for off-site VOC confirmation analysis at a frequency of 10 percent

QA split samples for all off-site samples sent to AML will be sent to ECB Laboratory at a frequency of 10 percent for analysis of the respective analyses.

Additional QC samples include one PE sample which will be analyzed by AML for VOCs in groundwater, as well as any QC samples required by the analytical method (i.e., LCS, laboratory duplicate). A detailed description of QC samples being collected is discussed in FSP Section 3.3. FSP Tables 5-2 through 5-6 and associated FSP Figures 5-1 through 5-8 depict the locations for these QA/QC samples to be collected.

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QAPP Worksheet #18
(UFP-QAPP Manual Section 3.1.1)
Sampling Locations and Methods/SOP Requirements Table

Sampling Location	Matrix	Sample Depth	Analytical Group	Concentration Level	Number of Samples[1] (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
Jet Engine Repair Hangar Aircraft Wash Area No. 2	Groundwater[2] (borehole monitoring well)	See FSP Section 5.1/Table 5-2	VOCs (off-site) Possible Natural Attenuation	Low	See FSP Section 5.1/Table 5-2	See FSP Section 5.1/Table 5-2	WP Section 6.1.3
Sewage Treatment Plant and Landfill	Surface Soil (0 – 6 feet)	See FSP Section 5.2/Table 5-3	SVOCs RCRA Metals (all off-site)	Low	See FSP Section 5.2/Table 5-3	See FSP Section 5.2/Table 5-3	WP Section 6.2.3
	Subsurface Soil (0.5 – 2 feet)		VOCs (off-site)				
	Subsurface Soil (2 – 10 feet)		VOCs SVOCs RCRA Metals (all off-site)				
	Subsurface Soil (10 feet – Groundwater)		VOCs (off-site)				
	Sediment		VOCs SVOCs RCRA Metals (all off-site)				
	Surface Water		VOCs SVOCs RCRA Metals (all off-site)				
	Groundwater[2] (borehole)		VOCs (on-site)				

Sampling Location	Matrix	Sample Depth	Analytical Group	Concentration Level	Number of Samples[(1)] (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
Sewage Treatment Plant and Landfill (continued)	Groundwater (Direct-Push)[3]	See FSP Section 5.2/Table 5-3	VOCs (on-site)	Low	See FSP Section 5.2/Table 5-3	See FSP Section 5.2/Table 5-3	WP Section 6.2.3
	Groundwater (Confirmation)[3]		VOCs (off-site)				
LOX Plant	Surface Soil (0 – 6 feet)	See FSP Section 5.3/Table 5-4	SVOCs (off-site)	Low	See FSP Section 5.3/Table 5-4	See FSP Section 5.3/Table 5-4	WP Section 6.3.3
	Subsurface Soil (0.5 – 2 feet)		VOCs (off-site)				
	Subsurface Soil (2 – 10 feet)		VOCs SVOCs (all off-site)				
	Subsurface Soil (10 feet – Groundwater)		VOCs (off-site)				
	Sediment		VOCs SVOCs (all off-site)				
	Surface Water		VOCs SVOCs (all off-site)				
	Groundwater (from borehole monitoring well)[2]		VOCs (off-site) Possible Natural Attenuation				

Sampling Location	Matrix	Sample Depth	Analytical Group	Concentration Level	Number of Samples[(1)] (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
LOX Plant (continued)	Groundwater (Direct-Push)[3]	See FSP Section 5.3/Table 5-4	VOCs (on-site)	Low	See FSP Section 5.3/Table 5-4	See FSP Section 5.3/Table 5-4	WP Section 6.3.3
	Groundwater (Confirmation)[3]		VOCs (off-site)				
Building 837 and Former Building 847	Groundwater [2] (from borehole monitoring well)	See FSP Section 5.4/Table 5-5	VOCs (off-site) Possible Natural Attenuation	Low	See FSP Section 5.4/Table 5-5	See FSP Section 5.4/Table 5-5	WP Section 6.4.3
	Surface Soil (0 - 6 feet)		SVOCs RCRA Metals (all off-site)				
	Subsurface Soil (0.5 – 2 feet)		VOCs (off-site)				
Groundwater[3]	Water	See FSP Section 5.5 Table 5-6	VOCs	Low	See FSP Section 5.5 Table 5-6	See FSP Section 5.5 Table 5-6	WP Sections noted above for specific area
Groundwater Monitoring Wells[4]	Water	NA	VOCs MEE Metals (Iron only) Anions Sulfide TOC	Low	To Be Determined	See FSP Section 5.5.1.3	WP Section 6.0

Sampling Location	Matrix	Sample Depth	Analytical Group	Concentration Level	Number of Samples[(1)] (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
See specific FSP section noted for planned sampling rationale and approach. Associated FSP Figures 5-1 through 5-8 depict the sample locations. [1]Number of samples will be determined based on the findings of the field investigation and/or field screening. [2]Groundwater samples collected from borehole from monitoring wells analyzed off-site for VOCs. These monitoring wells may also be sampled for natural attenuation indicator parameters (MEE, iron, anions, sulfide, and TOC). [3]Groundwater samples collected during direct-push activities will be analyzed on-site for selected VOCs using a field GC. Ten percent of these samples will be sent to an off-site lab for confirmation analysis of VOCs. [4]Groundwater samples collected from monitoring wells will be collected and analyzed off-site for VOCs. Twenty percent of these new monitoring wells will also be sampled for natural attenuation indicator parameters (MEE, iron, anions, sulfide, and TOC). Monitoring wells for natural attenuation indicator parameters will be selected once the configuration of the monitoring network in known.							

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QAPP Worksheet #19
(UFP-QAPP Manual Section 3.1.1)
Analytical SOP Requirements Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method[1] / SOP Reference[2]	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation / analysis)
Soil Sediment	VOCs	Low	SW-846 5035A/8260B	3 40-mL vials with Teflon-lined septa and open screw-caps, pre-weighed, and containing magnetic stir bars AND 1 container of sample filled with no headspace for determination of moisture content	Frozen (-7 ºC to -15 ºC) 4 ºC	14 Days Analyzed
	SVOCs	Low	SW-846 3550B/8270D	4-ounce clear wide-mouth glass jars with Teflon-lined septa caps	4 ºC	14 Days to Extraction/40 Days for Analysis following extraction
	Metals[3] (including mercury)	Low	SW-846 3050B/6020A SW-846 7471A (Mercury)	4-ounce clear wide-mouth glass jars with Teflon-lined septa caps	4 ºC	6 Months Analyzed (except Mercury) Mercury 28 Days Analyzed

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method[1] / SOP Reference[2]	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation / analysis)
Water	VOCs	Low	SW-846 5030B/8260B	3 x 40 mL glass Teflon-lined septa caps	No Headspace, HCl to pH <2, 4 ºC If sample effervesces, 4 ºC	14 Days (Preserved) 7 Days (Unpreserved) Analyzed
	SVOCs	Low	SW-846 3510B/8270D	2 x 1-Liter amber glass bottles Teflon-lined caps	4 ºC	7 Days to Extraction/40 Days for Analysis following extraction
	MEE	Low	RSK-175	3 x 40 mL glass Teflon-lined septa caps	No Headspace, HCl to pH <2, 4 ºC	14 Days Analyzed
	Metals[3] (including mercury)	Low	SW-846 3020A/6020A SW-846 7470A (Mercury)	500 mL HDPE	HNO_3 to pH < 2 4 ºC	6 Months Analyzed (except Mercury) Mercury 28 Days Analyzed
	Anions (Nitrate and Nitrite)	Low	EPA Method 300.0	1-250 mL HDPE	4 ºC	48 hours Analyzed
	Anions (Chloride and Sulfate)	Low	EPA Method 300.0	1-250 mL HDPE	4 ºC	28 Days Analyzed
	Sulfide	Low	EPA Method 376.1	1-Liiter HDPE	Zn acetate, NaOH to pH > 9, 4 ºC	7 Days Analyzed

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method[1] / SOP Reference[2]	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation / analysis)
Water	TOC	Low	SW-846 9060	1-125 mL HDPE	H_2SO_4 to pH < 2, 4 ºC	28 Days Analyzed

[1]When two methods are presented, the first method is the preparation method and the second method is the analytical method.
[2]Copies of AML's SOPs for each analysis are provided in Appendix A.
[3]Metals for the RI activities include RCRA Metals. Metals for the natural attenuation indicator parameters include iron.

ºC = Degrees Celsius
HCl = Hydrochloric Acid
HNO_3 = Nitric Acid
H_2SO_4 = Sulfuric Acid
NaOH = Sodium Hydroxide
HDPE = High Density Polyethylene

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QAPP Worksheet #20
(UFP-QAPP Manual Section 3.1.1)
Field Quality Control Sample Summary Table

The following table summarizes by matrix, analytical group, and concentration level the number of field QC samples that will be collected and submitted to the laboratory.

Matrix	Analytical Group	Conc. Level	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations[2]	No. of Field Duplicate Pairs[3]	No. of MS/MSD Samples	No. of Trip Blanks	No. of Rinsate Blanks[4]	No. of PE Samples	Total No. of Samples to Lab
Jet Engine Repair Hangar/Aircraft Wash Area No. 2										
Groundwater (borehole monitoring well)	VOCs Natural Attenuation[5]	Low	SW-846 8260B Natural Attenuation[5]	See FSP Section 5.1/ Table 5-2	NA	NA	1 per cooler	NA	0	See FSP Section 5.1/ Table 5-2
Sewage Treatment Plant and Landfill										
Surface Soil (0 – 6 feet)	SVOCs RCRA Metals	Low	SW-846 8270D SW-846 6020A/7471A	See FSP Section 5.2/ Table 5-3	1 per 10 samples	1 per 5 samples	NA	1 per 20 samples	0	See FSP Section 5.2/ Table 5-3
Subsurface Soil (0.5 – 2 feet)	VOCs		SW-846 8260B		1 per 10 samples	1 per 5 samples	NA	1 per 20 samples	0	
Subsurface Soil (2 – 10 feet)	VOCs SVOCs RCRA Metals		SW-846 8260B SW-846 8270D SW-846 6020A/7471A		1 per 10 samples	1 per 5 samples	NA	1 per 20 samples	0	
Surface Soil (10 feet – Groundwater)	VOCs		SW-846 8260B		1 per 10 samples	1 per 5 samples	NA	1 per 20 samples	0	
Sediment	VOCs SVOCs RCRA Metals		SW-846 8260B SW-846 8270D SW-846 6020A/7471A		1 per 10 samples	1 per 5 samples	NA	1 per 20 samples	0	
Surface Water	VOCs SVOCs RCRA Metals		SW-846 8260B SW-846 8270D SW-846 6020A/7470A		1 per 10 samples	1 per 5 samples	1 per cooler	1 per 20 samples	0	

Matrix	Analytical Group	Conc. Level	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations[2]	No. of Field Duplicate Pairs[3]	No. of MS/MSD Samples	No. of Trip Blanks	No. of Rinsate Blanks[4]	No. of PE Samples	Total No. of Samples to Lab
Sewage Treatment Plant and Landfill (continued)										
Groundwater (from borehole)	VOCs	Low	SW-846 8260B	See FSP Section 5.2/ Table 5-3	1 per 10 samples	1 per 5 samples	NA	1 per 20 samples	0	See FSP Section 5.2/ Table 5-3
Groundwater (Direct-Push)	VOCs		On-site VOC analysis with field GC		1 per 10 samples	NA	NA	1 per 20 samples	0	
Groundwater (Confirmation)	VOCs		SW-846 8260B		1 per 10 samples	1 per 5 samples	1 per cooler	1 per 20 samples	0	
LOX Plant										
Surface Soil (0 – 6 feet)	SVOCs	Low	SW-846 8270D	See FSP Section 5.3/Table 5-4	1 per 10 samples	1 per 5 samples	NA	1 per 20 samples	0	See FSP Section 5.3/Table 5-4
Subsurface Soil (0.5 – 2 feet)	VOCs		SW-846 8260B		1 per 10 samples	1 per 5 samples	NA	1 per 20 samples	0	
Subsurface Soil (2 – 10 feet)	VOCs SVOCs		SW-846 8260B SW-846 8270D		1 per 10 samples	1 per 5 samples	NA	1 per 20 samples	0	
Surface Soil (10 feet – Groundwater)	VOCs		SW-846 8260B		1 per 10 samples	1 per 5 samples	NA	1 per 20 samples	0	
Sediment	VOCs SVOCs		SW-846 8260B SW-846 8270D		1 per 10 samples	1 per 5 samples	NA	1 per 20 samples	0	
Surface Water	VOCs SVOCs		SW-846 8260B SW-846 8270D		1 per 10 samples	1 per 5 samples	1 per cooler	1 per 20 samples	0	

Matrix	Analytical Group	Conc. Level	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations[2]	No. of Field Duplicate Pairs[3]	No. of MS/MSD Samples	No. of Trip Blanks	No. of Rinsate Blanks[4]	No. of PE Samples	Total No. of Samples to Lab
LOX Plant (continued)										
Groundwater (from borehole monitoring well)	VOCs Natural Attenuation[5]	Low	SW-846 8260B Natural Attenuation[5]	See FSP Section 5.3/Table 5-4	1 per 10 samples	1 per 5 samples	NA	1 per 20 samples	0	See FSP Section 5.3/Table 5-4
Groundwater (Direct-Push)	VOCs		On-site VOC analysis with field GC		1 per 10 samples	1 per 5 samples	NA	1 per 20 samples	0	
Groundwater (Confirmation)	VOCs		SW-846 8260B		1 per 10 samples	1 per 5 samples	1 per cooler	1 per 20 samples	0	
Building 837 and Former Building 847										
Groundwater (from borehole monitoring well)	VOCs Natural Attenuation[5]	Low	SW-846 8260B Natural Attenuation[5]	See FSP Section 5.4/ Table 5-5	1 per 10 samples	1 per 5 samples	1 per cooler	1 per 20 samples	0	See FSP Section 5.4/ Table 5-5
Surface Soil (0 – 6 feet)	SVOCs RCRA Metals		SW-846 8270D SW-846 6020A/7471A		1 per 10 samples	1 per 5 samples	1 per cooler	1 per 20 samples	0	
Subsurface Soil (0.5 – 2 feet)	VOCs		SW-846 8260B		1 per 10 samples	1 per 5 samples	1 per cooler	1 per 20 samples	0	

Matrix	Analytical Group	Conc. Level	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations[2]	No. of Field Duplicate Pairs[3]	No. of MS/MSD Samples	No. of Trip Blanks	No. of Rinsate Blanks[4]	No. of PE Samples	Total No. of Samples to Lab
Groundwater Plumes (A, B, C, D, E)										
Groundwater (Direct-Push)	VOCs (on-site)	Low	On-site VOC analysis with field GC	See FSP Section 5.5/ Table 5-6	1 per 10 samples	1 per 5 samples	NA	1 per 20 samples	0	See FSP Section 5.5/ Table 5-6
Groundwater (Confirmation)	VOCs		SW-846 8260B		1 per 10 samples	1 per 5 samples	1 per cooler	1 per 20 samples	1	
Groundwater Monitoring Wells										
Groundwater	VOCs MEE Iron Anions Sulfide TOC	Low	SW-846 8260B RSK-175 SW-846 6020A EPA Method 300.0 EPA Method 376.1 SW-846 9060	To Be Determined	1 per 10 samples	1 per 5 samples	1 per cooler	1 per 20 samples	0	To Be Determined

[1]Copies of AML's SOPs for each analysis are provided in Appendix A.

[2]See FSP Tables 5-2 through 5-6 for number of soil and water samples to be collected at each specific area. The number of sample locations will be determined based upon the results of the field investigation activities. FSP Figures 5-1 through 5-8 depict the planned sample locations.

[3]In addition to field duplicate samples that will be submitted to the primary analysis lab, QA split samples will be sent to a secondary lab at the same frequency.

[4]Rinsate blanks will be collected when non-dedicated field equipment is used. Typically, rinsate blanks are collected at a frequency of 20 percent. Rinsate blanks will be collected at least once a week but no more than once per day.

[5]Borehole monitoring wells will be analyzed off-site for VOCs. These monitoring wells may also be sampled for natural attenuation indicator parameters including MEE, anions, sulfide, iron, and TOC.

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QAPP Worksheet #21
(UFP-QAPP Manual Section 3.1.2)
Project Sampling SOP References Table

A complete discussion of sampling procedures and requirements is provided in FSP Section 6.0 and the associated tables, figures and appendices. Procedures are presented within the text of the FSP and formalized SOP documents are not provided separately. Further discussion of sampling procedures and requirements will reference the appropriate section of the FSP that addresses the item.

Reference Number	Title, Revision Date and / or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
FSP Section 6.0	Soil Sampling Subsurface Soils – FSP Section 6.3 Surface Soils – FSP Section 6.4	BMcD	VOC soil sample collection by SW-846 5035A	N	Instructions for soil sample collection.
			Shelby tubes		
			Split-Barrel		
	Sediment Sampling FSP Section 6.5	BMcD	Wet Method	N	Instructions for sediment sample collection.
			Dry Method		
	Groundwater Sampling FSP Section 6.2	BMcD	Bailers	N	Instructions for groundwater sample collection.
			Passive-Diffusion Bags		
			Direct-Push		
	Surface Water Sampling FSP Section 6.6	BMcD	Sample containers	N	Instructions for surface water sample collection.
FSP Section 7.4.3	Chain of Custody Form	BMcD	Not Applicable	N	Instructions for completion of the COC form.
FSP Section 7.4.1	Sample Numbering System	BMcD	Not Applicable	N	Instructions for development of appropriate sample names.
FSP Section 7.2	Field Logbook	BMcD	Not Applicable	N	Instructions for completion of the field logbook.
FSP Section 7.2	Field Forms	BMcD	Not Applicable	N	Instructions for completion of field forms relevant to the project.

Reference Number	Title, Revision Date and / or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
FSP Section 8.0	Sample Handling and Shipment	BMcD	Not Applicable	N	Instructions for preparation of samples for shipment to the lab.
WP Section 6.0	Sample Location Rationale	BMcD	Not Applicable	N	Rationale and approach to determine sampling locations.

Equipment/Sample Containers Cleaning and Decontamination Procedure

During field investigations, various activities such as direct-push soil/groundwater sampling, soil boring, temporary piezometer, and monitoring well installation, surface/subsurface soil sampling, groundwater sampling, and decontamination of environmental equipment and investigative personnel will produce solid and/or liquid investigation-derived waste (IDW). The containerization, labeling, storage, evaluation, and disposal of the IDW are discussed in FSP Section 9.0.

The laboratory is responsible for cleaning and decontamination procedures for their equipment. These procedures are discussed in AML's *QAP* and/or SOPs (see Appendix A).

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QAPP Worksheet #22
(UFP-QAPP Manual Section 3.1.2.4)
Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Calibration Activity	Maint. Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference
Photoionization Detector (PID)	See Operation Manual, Appendix C							FSM	Organic Vapor Meter (OVM) / Datalogger Instruction Manual
Hach® YSI 556 Multi-probe system (pH, temperature, specific conductivity, and dissolved oxygen)	See Operation Manual, Appendix C							FSM	YSI 556 Multi probe system Operations Manual
Hach® 2100 Turbidity Meter	See Operation Manual Appendix C							FSM	Portable Turbidmeter Model 2100
CHEMets® Kit (Ferrous iron meter)	See Operation Manual Appendix C							FSM	Iron CHEMets® Ferrous Iron Procedure
GC	See Field Procedures Manual, Appendix B							EPS	See *FPM*
Operation Manuals and further instructions for use can be found online for the YSI and turbidity meter at http://www.geotechenv.com. Operations Manuals and further instructions for use for the ferrous iron meter at http://www.chemetrics.com									

Field Equipment Inspection and Acceptance Procedures

Field equipment used for field monitoring (PID, ferrous iron, etc) and stability measurements (pH, temperature, etc.) will be used during field investigations. With the exception of the on-site GC, the FSM will be responsible for insuring that these instruments are calibrated daily. These instruments will be calibrated prior to sample collection and again at the end of the day after all samples are collected. The inspection, calibration, and maintenance of the on-site GC instrument will be the responsibility of EPS. If problems occur with the field instruments, which cannot be resolved by the field team member, the FSM will be notified. If field equipment fails inspection, it the FSM's responsibility to investigate and resolve the problem. Any problems will be noted in the field logbook and prolonged time spent investigating and solving the equipment problem(s) will be noted and reported to the Project Manager. If necessary, the FSM should contact the distributor of the equipment for instructions and/or replacement.

Field Operations Documentation Procedures

Field operations documentation procedures are presented in FSP Section 7.0.

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QAPP Worksheet #23
(UFP-QAPP Manual Section 3.2.1)
Analytical SOP References Table

Soil, sediment, and water samples will be collected and submitted to the primary laboratory (AML) and ECB Laboratory for analysis. Soil and sediment samples will be analyzed for VOCs, SVOCs, and/or RCRA metals. Surface water samples will be analyzed for VOCs, SVOCs, and/or RCRA metals. Groundwater samples collected from borehole monitoring wells will be analyzed off-site for VOCs and possibly natural attenuation indicator parameters (MEE, iron, anions, sulfide, and TOC), while groundwater samples collected direct-push activities will be analyzed on-site for select VOCs using a field GC. Ten percent of the samples analyzed on-site will be sent off-site to AML for confirmation analysis of VOCs. Groundwater samples collected after installation of the monitoring wells will be collected for VOCs as well as natural attenuation indicator parameters including MEE, iron, anions, sulfide, and TOC. Specific analytical method and the respective laboratory SOPs are noted below. AML's SOPs are provided in Appendix A.

Reference Number	Title, Revision Date, and / or Number[a]	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
O-BNA-SVOCs-RD	Base Neutral and Acid (BNA) Semivolatile Organic Compounds (SVOCs) by Gas Chromatography/Mass Spectrometry (GC/MS) (EPA 625 and SW-846 8270D). Prepared by AML (October 28, 2005).	Definitive	SVOC	Gas Chromatograph/ Mass Spectrometer (GC/MS)	AML	N
O-VOCs-GCMS-RC	Volatile Organic Compounds in Water and Soil by Gas Chromatography/Mass Spectrometry (GC/MS). Prepared by AML (October 28, 2005).	Definitive	VOC	GC/MS	AML	N

Reference Number	Title, Revision Date, and / or Number[a]	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
O-SoilVOCs-5035A-RC	Soil Sample Collection and Preservation Procedures for the Analysis of VOCs by SW-846 Draft Method 5035A. Prepared by AML (October 28, 2005).	Definitive	VOC	Not Applicable	AML	N
O-MEE-RC	Analysis of Methane, Ethane, and Ethene (MEE) in Aqueous Samples by Headspace Sampling and Gas Chromatography/FID (GC/FID) (EPA RSK-175 and EPA SW846-8015B mod.) Prepared by AML. (October 28, 2005).	Definitive	MEE	GC	AML	N
I-3020APrep-RD	Acid Digestion of Aqueous Samples for Total Recoverable Metals Analysis by GFAA or ICP-MS. Prepared by AML (October 28, 2005).	NA	Metals - Prep	Not Applicable	AML	N
I-3050BPrep-RD	Acid Digestion of Soils, Sediments, and Sludges for Total Recoverable Metals Analysis. Prepared by AML (October 28, 2005).	NA	Metals - Prep	Not Applicable	AML	N
I-ICP-MS-Metals-RD	Metals Analysis by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) (EPA 200.8 and SW-846 6020A). Prepared by AML (October 21, 2005).	Definitive	Metal	ICP-MS (Inductively Coupled Plasma-Mass Spectrometry)	AML	N

Reference Number	Title, Revision Date, and / or Number[a]	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
I-Mercury-CVAA-RD	Mercury in Soil and Water by Cold Vapor Atomic Absorption (CVAA) Technique (EPA 245.1, 245.2, 245.5, SW-846 7470A, and 7471B). Prepared by AML (October 28, 2005).	Definitive	Metal	Cold Vapor Atomic Absorption (CVAA)	AML	N
I-Anions-IC-RC	Determination of Inorganic Anions by Ion Chromatography (EPA 300.0, 300.1, and SW-846 9056A). Prepared by AML (October 28, 2005).	Definitive	Inorganics	Ion Chromatography (IC)	AML	N
I-Sulfide-Titrimetry-RC	Sulfide by Titrimetry (EPA 376.1, SM 4500, and SW-846 9034). Prepared by AML (November 2, 2005).	Definitive	Wet Chemistry	NA	AML	N
I-TOC-RC	Total Organic Carbon. Prepared by AML (November 2, 2005).	Definitive	Wet Chemistry	Apollo 9000 TOC Combustion Analyzer	AML	N
Non-Analytical SOPs used by Laboratory in Support of Analysis						
Q-Cleaning-RD	Glassware Cleaning Procedures. Prepared by AML (October 31, 2005).	Not Applicable	Not Applicable	Not Applicable	AML	N
Q-Manual Integration-RD	Manual Integration. Prepared by AML (March 6, 2006).	Not Applicable	Not Applicable	Not Applicable	AML	N
Q-Manual Integration-Appendix.RD	Appendix to the SOP on Manual Integration. Prepared by AML (March 7, 2006).	Not Applicable	Not Applicable	Not Applicable	AML	N
Q-Water-RD	Reagent Water. Prepared by AML (October 31, 2005).	Not Applicable	Not Applicable	Not Applicable	AML	N

Reference Number	Title, Revision Date, and / or Number[a]	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
Q-Waste Management-RE	Waste Management and Disposal Including Sample Disposal. Prepared by AML (October 31, 2005).	Not Applicable	Not Applicable	Not Applicable	AML	N
Q-Temperature Control-RC	Temperature Measurements and Control of Ovens, Coolers, Refrigerators, and Freezers. Prepared by AML (October 11, 2005).	Not Applicable	Not Applicable	Not Applicable	AML	N
Q-Standards-RC	Analytical Standards and Reagents. Prepared by AML (October 31, 2005).	Not Applicable	Not Applicable	Not Applicable	AML	N
Q-MDL-LLR-RL-MQL-RC	Instrument Detection Limits (IDL), Method Detection Limits (MDL), Lowest Level for Reporting (LLR), Reporting Limits (RL), Method Quantitation Limits (MQL), and Initial Demonstration of Performance (IDP). Prepared by AML (October 31, 2005).	Not Applicable	Not Applicable	Not Applicable	AML	N
Q-Volumetric-RC	Volumetric Measurements and Control of Volume Measurement Devices. Prepared by AML (October 31, 2005).	Not Applicable	Not Applicable	Not Applicable	AML	N

Analytical SOP information for the on-site GC analysis for VOCs can be found in EPS' *FPM* (Appendix B).

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QAPP Worksheet #24
(UFP-QAPP Manual Section 3.2.2)
Analytical Instrument Calibration Table

The laboratory is responsible for analytical instrument calibration procedures. These procedures are expected to meet any requirements established in the applicable method (i.e., SW-846 8260B, SW-846 8270D, SW-846 6020A). A general discussion of AML's calibration procedures and frequency is presented in AML's *QAP* (see Appendix A), and specific requirements are detailed in AML's respective SOPs noted below and are provided in Appendix A. Tables 24-1 through 24-8 present the QC check procedures, frequency, acceptance criteria, and corrective actions for each analytical method/instrument.

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS	See specific SOPs and /or Section 9.4.4 in the AML *QAP*	See Section 9.4.4 in the AML *QAP*	See Section 9.4.4 in the AML *QAP*	See Section 9.4.4 in the AML *QAP*	AML	O-BNA-SVOCs-RD O-VOCs-GCMS-RC
GC	See specific SOP and/or Section 9.4.3 in the AML *QAP*	See Section 9.4.3 in the AML *QAP*	See Section 9.4.3 in the AML *QAP*	See Section 9.4.3 in the AML *QAP*	AML	O-MEE-RC
ICP-MS	See specific SOP and /or Section 9.4.1 in the AML *QAP*	See Section 9.4.1 in the AML *QAP*	See Section 9.4.1 in the AML *QAP*	See Section 9.4.1 in the AML *QAP*	AML	I-ICP-MS-Metals-RC
AA Spectrometer	See specific SOP and/or Section 9.4.1.3 in the AML *QAP*	See Section 9.4.1.3 in the AML *QAP*	See Section 9.4.1.3 in the AML *QAP*	See Section 9.4.1.3 in the AML *QAP*	AML	I-Mercury-CVAA-RD
Ion Chromatograph	See specific SOP and/or Section 9.4.2.1 in the AML *QAP*	See Section 9.4.2.1 in the AML *QAP*	See Section 9.4.2.1 in the AML *QAP*	See Section 9.4.2.1 in the AML *QAP*	AML	I-Anions-IC-RC

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
TOC Combustion Analyzed	See specific SOP and/or Section 9.4.2.6 in the AML *QAP*	See Section 9.4.2.6 in the AML *QAP*	See Section 9.4.2.6 in the AML *QAP*	See Section 9.4.2.6 in the AML *QAP*	AML	I-TOC-RC
Field GC	See *FPM* Appendix B	Daily/Continuously between sample analysis	See *FPM* Appendix B	See *FPM* Appendix B	EPS	See *FPM* Appendix B
Analytical SOP information for the on-site GC analysis for VOCs can be found in EPS' *FPM* (Appendix B).						

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QAPP Worksheet #25
(UFP-QAPP Manual Section 3.2.3)
Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

The laboratories are responsible for analytical instrument and equipment maintenance, testing, and inspection procedures. A general discussion of AML's instrument and equipment maintenance, testing, and inspection procedures is presented in AML's *QAP* (see Appendix A), and specific requirements are detailed in AML's respective SOPs noted on Worksheet #24. SOPs have been developed by AML to address instrument maintenance issues and are provided in Appendix A.

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
See list of instruments given in Worksheet #24	See specific SOP and /or Section 10 in the AML QAP	See specific SOP and /or Section 10 in the AML QAP	See specific SOP and /or Section 10 in the AML QAP	See specific SOP and /or Section 10 in the AML QAP	See specific SOP and /or Section 10 in the AML QAP	See specific SOP and /or Section 10 in the AML QAP	See specific SOP and /or Section 10 in the AML QAP	See specific SOP reference listed for the respective instrument listed on Worksheet #24
Analytical SOP information for the on-site GC analysis for VOCs can be found in EPS' *FPM* (Appendix B).								

Analytical Supply Inspection and Acceptance Procedures

The laboratories are responsible for maintaining procedures for inspection and acceptance of supplies, standards, and materials into their facility. These procedures are discussed in AML's *QAP* and SOPs (see Appendix A). The FSM will inspect sample containers, rental instruments, and other supplies for correct quantity and accuracy. The FSM is responsible for arranging for replacement of missing or damaged items.

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QAPP Worksheet #26
(UFP-QAPP Manual Appendix A)
Sample Handling System

This worksheet identifies components of the project-specific sample handling system. Personnel who are primarily responsible for ensuring proper handling, custody, and storage of field samples from the time of collection, to laboratory delivery, to final sample disposal are presented on this worksheet. In addition, the number of days that field samples and their extracts/digestates will be archived prior to disposal is provided.

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Field Sampling Team, BMcD
Sample Packaging (Personnel/Organization): Field Sampling Team, BMcD
Coordination of Shipment (Personnel/Organization): FSM, BMcD
Type of Shipment/Carrier: Overnight/FedEx
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Custodian, AML
Sample Custody and Storage (Personnel/Organization): Sample Custodian, AML
Sample Preparation (Personnel/Organization): Team Leader (VOC/SVOC, Wet Chemistry, Metals), AML
Sample Determinative Analysis (Personnel/Organization): Team Leader (VOC/SVOC, Wet Chemistry, Metals), AML
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): Samples will not be stored in the field, but will be shipped with 24 hours of collection. If in an emergency, they are stored in the field, they will be kept within the sample preservation range of 2-6 ºC.
Sample Extract/Digestate Storage (No. of days from extraction/digestion): Sample extraction and digestion must be conducted according to the analytical methods and the requirements given in Worksheet #19.
Biological Sample Storage (No. of days from sample collection): Not Applicable
SAMPLE DISPOSAL
Personnel/Organization: Sample Custodian, AML
Number of Days from Analysis: 20 Days following receipt of final data package.

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QAPP Worksheet #27
(UFP-QAPP Manual Section 3.3.3)
Sample Custody Requirements Table

A complete discussion of sample collection documentation, handling, tracking, and custody procedures and requirements is provided in FSP Sections 7.0 (Field Operations Documentation) and 8.0 (Sample Packaging and Shipping Requirements) and their associated appendices. General procedures are presented within the text of the FSP. Laboratory-specific procedures are provided in AML's SOP Q-SampleMgmt-RC (Appendix A). Further discussion of sample collection documentation, handling, tracking, and custody procedures and requirements can be referenced in the appropriate section of the FSP that addresses the item.

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to the laboratory):

The following sections in the FSP present the field sampling custody procedures:

7.4.3 Chain-of-Custody Records
7.4.4 Custody Seals
8.0 Sample Handling and Shipment

Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal):

AML's custody procedures are discussed in the laboratory's *QAP* and associated SOPs. A copy AML's *QAP* is provided in Appendix A.

Sample Identification Procedures:

The following sample documentation sections in FSP Section 7.4 present the sample identification procedures:

7.4.1 Sample Numbering System
- 7.4.1.1.1 Sample Group
- 7.4.1.1.2 Sample Designator
- 7.4.1.1.3 QA/QC Designators

Chain-of-Custody Procedures:

The following section in the FSP presents the COC procedures:

7.4.3 Chain-of-Custody Records

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QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)
QC Samples Table

QAPP Worksheet #28-1 VOC analysis using SW-846 8260B (Soil/Sediment)

Matrix	Soil/Sediment
Analytical Group	VOCs
Concentration Level	Low
Sampling SOP	Subsurface Soil FSP Section 6.3, Surface Soil FSP Section 6.4, Sediment FSP Section 6.5
Analytical Method / SOP Reference	SW-846 8260B
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Tables 5-2 through 5-5

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per Batch (20 samples)	No target compounds > ½ PQL or >PQL for common contaminants	See O-VOCs-GCMS-RC	Analyst	Sensitivity Accuracy/Bias – Contamination	No target compounds > ½ PQL or >PQL for common contaminants
LCS	1 per Batch (20 samples)	See Table 12-3	See O-VOCs-GCMS-RC	Analyst	Accuracy/Bias	REC within limits[1]
MS/MSD Samples	5% of field samples (1 site-specific per Batch)	See Table 12-3	See O-VOCs-GCMS-RC	Analyst	Accuracy/Bias/ Precision	REC within limits[1] RPD within limits[1]
Surrogate Spike	Surrogates are spiked into each sample	See Table 12-11	See O-VOCs-GCMS-RC	Analyst	Accuracy/Bias	REC within limits

Matrix	Soil/Sediment
Analytical Group	VOCs
Concentration Level	Low
Sampling SOP	Subsurface Soil FSP Section 6.3, Surface Soil FSP Section 6.4, Sediment FSP Section 6.5
Analytical Method / SOP Reference	SW-846 8260B
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Tables 5-2 through 5-5

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Rinsate Blank	20% of field samples; Once a week but no more than once a day (For use of non-dedicated equipment)	No target compounds > ½ PQL or >PQL for common contaminants	Investigate source of contamination.	BMcD Chemist	Sensitivity	No target compounds > ½ PQL or >PQL for common contaminants
Field Duplicate	10% of field samples	See Table 12-1	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Precision	See Table 12-1
QA Split Sample	10% of field samples	See Table 12-1	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Comparability Precision	See Table 12-1
Not Applicable	Each Sample	Sample analyzed with the applicable method holding time	Reject Data	BMcD Chemist	Representativeness	Sample analyzed with the applicable method holding time

[1]The DoD QSM also has provision for acceptance of a limited number of sporadic MEs for analyses that contain greater than 11 parameters. Based on the VOC analyte list provided in Table 12-3, up to three (3) sporadic MEs are allowed before corrective actions are required.

QAPP Worksheet #28-2 VOC analysis using SW-846 8260B (Water)

Matrix	Water
Analytical Group	VOCs
Concentration Level	Low
Sampling SOP	Groundwater FSP Section 6.2, Surface Water FSP Section 6.6,
Analytical Method / SOP Reference	SW-846 8270D
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Tables 5-2 through 5-6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per Batch (20 samples)	No target compounds > ½ PQL or >PQL for common contaminants	See O-VOCs-GCMS-RC	Analyst	Sensitivity Accuracy/Bias – Contamination	No target compounds > ½ PQL or >PQL for common contaminants
LCS	1 per Batch (20 samples)	See Table 12-4	See O-VOCs-GCMS-RC	Analyst	Accuracy/Bias	REC within limits[1]
MS/MSD Samples	5% of field samples (1 site-specific per Batch)	See Table 12-4	See O-VOCs-GCMS-RC	Analyst	Accuracy/Bias/ Precision	REC within limits[1] RPD within limits[1]
Surrogate Spike	Surrogates are spiked into each sample	See Table 12-11	See O-VOCs-GCMS-RC	Analyst	Accuracy/Bias	REC within limits
Trip Blank	One submitted with each day's VOC samples	No target compounds > ½ PQL or >PQL for common contaminants	Investigate source of contamination.	BMcD Chemist	Accuracy/Bias	No target compounds > ½ PQL or >PQL for common contaminants
Rinsate Blank	20% of field samples; Once a week but no more than once a day (For use of non-dedicated equipment)	No target compounds > ½ PQL or >PQL for common contaminants	Investigate source of contamination.	BMcD Chemist	Sensitivity	No target compounds > ½ PQL or >PQL for common contaminants

Matrix	Water
Analytical Group	VOCs
Concentration Level	Low
Sampling SOP	Groundwater FSP Section 6.2, Surface Water FSP Section 6.6,
Analytical Method / SOP Reference	SW-846 8270D
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Tables 5-2 through 5-6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Duplicate	10% of field samples	See Table 12-2	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Precision	See Table 12-2
QA Split Sample	10% of field samples	See Table 12-2	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Comparability Precision	See Table 12-2
PE Sample	Once	See Worksheet #12 "PE Sample"	Evaluation of discrepancies between results, possible review of raw data.	BMcD Chemist	Comparability Precision	See Table 12-2
Confirmation Sample	10% of field samples	Linear Regression (r^2) ≥ 0.70 See Worksheet #12 "Confirmation Samples".	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Comparability Precision	See Worksheet #12 (Coefficient correlation test)
Not Applicable	Each Sample	Sample analyzed with the applicable method holding time	Reject Data	BMcD Chemist	Representativeness	Sample analyzed with the applicable method holding time

[1]The DoD QSM also has provision for acceptance of a limited number of sporadic MEs for analyses that contain greater than 11 parameters. Based on the VOC analyte list provided in Table 12-4, up to three (3) sporadic MEs are allowed before corrective actions are required.

QAPP Worksheet #28-3 SVOC analysis using SW-846 8270D (Soil/Sediment)

Matrix	Soil/Sediment
Analytical Group	SVOCs
Concentration Level	Low
Sampling SOP	Subsurface Soil FSP Section 6.3, Surface Soil FSP Section 6.4, Sediment FSP Section 6.5
Analytical Method / SOP Reference	SW-846 8270C
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Tables 5-2 through 5-5

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per Batch (20 samples)	No target compounds > ½ PQL or >PQL for common contaminants	See O-BNA-SVOCs-RD	Analyst	Sensitivity Accuracy/Bias – Contamination	No target compounds > ½ PQL or >PQL for common contaminants
LCS	1 per Batch (20 samples)	See Table 12-5	See O-BNA-SVOCs-RD	Analyst	Accuracy/Bias	REC within limits[1]
MS/MSD	5% of field samples (1 site-specific per Batch)	See Table 12-5	See O-BNA-SVOCs-RD	Analyst	Accuracy/Bias/ Precision	REC within limits[1] RPD within limits[1]
Surrogate Spike	Surrogates are spiked into each sample	See Table 12-12	See O-BNA-SVOCs-RD	Analyst	Accuracy/Bias	REC within limits
Rinsate Blank	20% of field samples; Once a week but no more than once a day (For use of non-dedicated equipment)	No target compounds > ½ PQL or >PQL for common contaminants	Investigate source of contamination.	BMcD Chemist	Sensitivity	No target compounds > ½ PQL or >PQL for common contaminants
Field Duplicate	10% of field samples	See Table 12-1	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Precision	See Table 12-1

Matrix	Soil/Sediment
Analytical Group	SVOCs
Concentration Level	Low
Sampling SOP	Subsurface Soil FSP Section 6.3, Surface Soil FSP Section 6.4, Sediment FSP Section 6.5
Analytical Method / SOP Reference	SW-846 8270C
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Tables 5-2 through 5-5

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
QA Split Sample	10% of field samples	See Table 12-1	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Comparability Precision	See Table 12-1
Not Applicable	Each Sample	Sample analyzed with the applicable method holding time	Reject Data	BMcD Chemist	Representativeness	Sample analyzed with the applicable method holding time

[1]The DoD QSM also has provision for acceptance of a limited number of sporadic MEs for analyses that contain greater than 11 parameters. Based on the SVOC analyte list provided in Tables 12-5, up to three (3) sporadic MEs are allowed before corrective actions are required.

QAPP Worksheet #28-4 SVOC analysis using SW-846 8270D (Water)

Matrix	Water
Analytical Group	SVOCs
Concentration Level	Low
Sampling SOP	Groundwater FSP Section 6.2, Surface Water FSP Section 6.6
Analytical Method / SOP Reference	SW-846 8270D
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Tables 5-2 through 5-6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per Batch (20 samples)	No target compounds > ½ PQL or >PQL for common contaminants	See O-BNA-SVOCs-RD	Analyst	Sensitivity Accuracy/Bias – Contamination	No target compounds > ½ PQL or >PQL for common contaminants
LCS	1 per Batch (20 samples)	See Table 12-6	See O-BNA-SVOCs-RD	Analyst	Accuracy/Bias	REC within limits[1]
MS/MSD	5% of field samples (1 site-specific per Batch)	See Table 12-6	See O-BNA-SVOCs-RD	Analyst	Accuracy/Bias/ Precision	REC within limits[1] RPD within limits[1]
Surrogate Spike	Surrogates are spiked into each sample	See Table 12-12	See O-BNA-SVOCs-RD	Analyst	Accuracy/Bias	REC within limits
Rinsate Blank	20% of field samples; Once a week but no more than once a day (For use of non-dedicated equipment)	No target compounds > ½ PQL or >PQL for common contaminants	Investigate source of contamination.	BMcD Chemist	Sensitivity	No target compounds > ½ PQL or >PQL for common contaminants
Field Duplicate	10% of field samples	See Table 12-2	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Precision	See Table 12-2

Matrix	Water
Analytical Group	SVOCs
Concentration Level	Low
Sampling SOP	Groundwater FSP Section 6.2, Surface Water FSP Section 6.6
Analytical Method / SOP Reference	SW-846 8270D
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Tables 5-2 through 5-6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
QA Split Sample	10% of field samples	See Table 12-2	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Comparability Precision	See Table 12-2
Not Applicable	Each Sample	Sample analyzed with the applicable method holding time	Reject Data	BMcD Chemist	Representativeness	Sample analyzed with the applicable method holding time

[1]The DoD QSM also has provision for acceptance of a limited number of sporadic MEs for analyses that contain greater than 11 parameters. Based on the SVOC analyte list provided in Tables 12-6, up to three (3) sporadic MEs are allowed before corrective actions are required.

QAPP Worksheet #28-5 MEE analysis using RSK-175 (Water)

Matrix	Water
Analytical Group	MEE
Concentration Level	Low
Sampling SOP	Groundwater FSP Section 6.2
Analytical Method / SOP Reference	RSK-175
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Table 5-6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per Batch (20 samples)	No target compounds > ½ PQL	See O-MEE-RC	Analyst	Sensitivity Accuracy/Bias – Contamination	No target compounds > ½ PQL
LCS	1 per Batch (20 samples)	See Table 12-7	See O-MEE-RC	Analyst	Accuracy/Bias	REC within limits
MS/MSD Samples	5% of field samples (1 site-specific per Batch)	See Table 12-7	See O-MEE-RC	Analyst	Accuracy/Bias/ Precision	REC within limits RPD within limits
Trip Blank	One submitted with each day's MEE samples	No target compounds > ½ PQL	If detections > PQL are noted, the lab will be asked to confirm the results if similar detections are noted in the field samples.	BMcD Chemist	Accuracy/Bias	No target compounds > ½ PQL
Rinsate Blank	20% of field samples; Once a week but no more than once a day (For use of non-dedicated equipment)	No target compounds > ½ PQL	Investigate source of contamination.	BMcD Chemist	Sensitivity	No target compounds > ½ PQL
Field Duplicate	10% of field samples	See Table 12-2	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Precision	See Table 12-2

Matrix	Water
Analytical Group	MEE
Concentration Level	Low
Sampling SOP	Groundwater FSP Section 6.2
Analytical Method / SOP Reference	RSK-175
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Table 5-6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
QA Split Sample	10% of field samples	See Table 12-2	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Comparability Precision	See Table 12-2
Not Applicable	Each Sample	Sample analyzed with the applicable method holding time	Reject Data	BMcD Chemist	Representativeness	Sample analyzed with the applicable method holding time

QAPP Worksheet #28-6 Metals analysis using SW-846 6020A (Soil/Sediment)

Matrix	Soil/Sediment
Analytical Group	Metals
Concentration Level	Low
Sampling SOP	Subsurface Soil FSP Section 6.3, Surface Soil FSP Section 6.4, Sediment FSP 6.5
Analytical Method / SOP Reference	Metals SW-846 6020A
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Tables 5-2 through 5-5

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per Batch (20 samples)	No target compounds > ½ PQL	See I-ICP-MS-Metals-RC	Analyst	Sensitivity Accuracy/Bias – Contamination	No target compounds > ½ PQL
LCS	1 per Batch (20 samples)	See Table 12-8	See I-ICP-MS-Metals-RC	Analyst	Accuracy/Bias	REC within limits
MS/MSD Samples	5% of field samples (1 site-specific per Batch)	See Table 12-18	See I-ICP-MS-Metals-RC	Analyst	Accuracy/Bias/ Precision	REC within limits RPD within limits
Rinsate Blank	20% of field samples; Once a week but no more than once a day (For use of non-dedicated equipment)	No target compounds > ½ PQL	Investigate source of contamination.	BMcD Chemist	Sensitivity	No target compounds > ½ PQL
Field Duplicate	10% of field samples	See Table 12-1	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Precision	See Table 12-1
QA Split Sample	10% of field samples	See Table 12-1	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Comparability Precision	See Table 12-1

Matrix	Soil/Sediment
Analytical Group	Metals
Concentration Level	Low
Sampling SOP	Subsurface Soil FSP Section 6.3, Surface Soil FSP Section 6.4, Sediment FSP 6.5
Analytical Method / SOP Reference	Metals SW-846 6020A
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Tables 5-2 through 5-5

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Not Applicable	Each Sample	Sample analyzed with the applicable method holding time	Reject Data	BMcD Chemist	Representativeness	Sample analyzed with the applicable method holding time

QAPP Worksheet #28-7 Mercury analysis using SW-846 7471A (Soil/Sediment)

Matrix	Soil/Sediment
Analytical Group	Mercury
Concentration Level	Low
Sampling SOP	Subsurface Soil FSP Section 6.3, Surface Soil FSP Section 6.4, Sediment FSP 6.5
Analytical Method / SOP Reference	SW-846 7471A
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Tables 5-2 through 5-5

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per Batch (20 samples)	No target compounds > ½ PQL	See I-Mercury-CVAA-RD	Analyst	Sensitivity Accuracy/Bias – Contamination	No target compounds > ½ PQL
LCS	1 per Batch (20 samples)	See Table 12-8	See I-Mercury-CVAA-RD	Analyst	Accuracy/Bias	REC within limits
MS/MSD Samples	5% of field samples (1 site-specific per Batch)	See Table 12-8	See I-Mercury-CVAA-RD	Analyst	Accuracy/Bias/ Precision	REC within limits RPD within limits
Rinsate Blank	20% of field samples; Once a week but no more than once a day (For use of non-dedicated equipment)	No target compounds > ½ PQL	Investigate source of contamination.	BMcD Chemist	Sensitivity	No target compounds > ½ PQL
Field Duplicate	10% of field samples	See Table 12-1	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Precision	See Table 12-1

Matrix	Soil/Sediment
Analytical Group	Mercury
Concentration Level	Low
Sampling SOP	Subsurface Soil FSP Section 6.3, Surface Soil FSP Section 6.4, Sediment FSP 6.5
Analytical Method / SOP Reference	SW-846 7471A
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Tables 5-2 through 5-5

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
QA Split Sample	10% of field samples	See Table 12-1	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Comparability Precision	See Table 12-1
Not Applicable	Each Sample	Sample analyzed with the applicable method holding time	Reject Data	BMcD Chemist	Representativeness	Sample analyzed with the applicable method holding time

QAPP Worksheet #28-8 Metals analysis using SW-846 Methods 6020A (Water)

Matrix	Water
Analytical Group	Metals
Concentration Level	Low
Sampling SOP	Groundwater FSP Section 6.2, Surface Water FSP Section 6.4
Analytical Method / SOP Reference	SW-846 6020A
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Tables 5-2 through 5-6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per Batch (20 samples)	No target compounds > ½ PQL	I-ICP-MS Metals-RC	Analyst	Sensitivity Accuracy/Bias – Contamination	No target compounds > ½ PQL
LCS	1 per Batch (20 samples)	See Table 12-9	I-ICP-MS Metals-RC	Analyst	Accuracy/Bias	REC within limits
MS/MSD Samples	5% of field samples (1 site-specific per Batch)	See Table 12-9	I-ICP-MS Metals-RC	Analyst	Accuracy/Bias/ Precision	REC within limits RPD within limits
Rinsate Blank	20% of field samples; Once a week but no more than once a day (For use of non-dedicated equipment)	No target compounds > ½ PQL	Investigate source of contamination.	BMcD Chemist	Sensitivity	No target compounds > ½ PQL
Field Duplicate	10% of field samples	See Table 12-2	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Precision	See Table 12-2

Matrix	Water
Analytical Group	Metals
Concentration Level	Low
Sampling SOP	Groundwater FSP Section 6.2, Surface Water FSP Section 6.4
Analytical Method / SOP Reference	SW-846 6020A
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Tables 5-2 through 5-6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
QA Split Sample	10% of field samples	See Table 12-2	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Comparability Precision	See Table 12-2
Not Applicable	Each Sample	Sample analyzed with the applicable method holding time	Reject Data	BMcD Chemist	Representativeness	Sample analyzed with the applicable method holding time

QAPP Worksheet #28-9 Mercury analysis using SW-846 7470A (Water)

Matrix	Water
Analytical Group	Mercury
Concentration Level	Low
Sampling SOP	Surface Water FSP Section 6.4
Analytical Method / SOP Reference	SW-846 7470A
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Tables 5-2 through 5-6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per Batch (20 samples)	No target compounds > ½ PQL	See I-Mercury-CVAA-RD	Analyst	Sensitivity Accuracy/Bias – Contamination	No target compounds > ½ PQL
LCS	1 per Batch (20 samples)	See Table 12-9	See I-Mercury-CVAA-RD	Analyst	Accuracy/Bias	REC within limits
MS/MSD Samples	5% of field samples (1 site-specific per Batch)	See Table 12-9	See I-Mercury-CVAA-RD	Analyst	Accuracy/Bias/ Precision	REC within limits RPD within limits
Rinsate Blank	20% of field samples; Once a week but no more than once a day (For use of non-dedicated equipment)	No target compounds > ½ PQL	Investigate source of contamination.	BMcD Chemist	Sensitivity	No target compounds > ½ PQL
Field Duplicate	10% of field samples	See Table 12-2	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Precision	See Table 12-2
QA Split Sample	10% of field samples	See Table 12-2	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Comparability Precision	See Table 12-2
Not Applicable	Each Sample	Sample analyzed with the applicable method holding time	Reject Data	BMcD Chemist	Representativeness	Sample analyzed with the applicable method holding time

QAPP Worksheet #28-10 Anions analysis using EPA Method 300.0 (Water)

Matrix	Water
Analytical Group	Anions (Chloride, Nitrate, Nitrite, Sulfate)
Concentration Level	Low
Sampling SOP	Groundwater FSP Section 6.2
Analytical Method / SOP Reference	EPA Method 300.0
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Table 5-6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per Batch (20 samples)	No target compounds > ½ PQL	See I-Anions-IC-RC	Analyst	Sensitivity Accuracy/Bias – Contamination	No target compounds > ½ PQL
LCS	1 per Batch (20 samples)	Within laboratory acceptance limits. See Table 12-10	See I-Anions-IC-RC	Analyst	Accuracy/Bias	REC within limits
MS/MSD Samples	5% of field samples (1 site-specific per Batch)	Within laboratory acceptance limits. See Table 12-10	See I-Anions-IC-RC	Analyst	Accuracy/Bias/ Precision	REC within limits RPD within limits
Rinsate Blank	20% of field samples; Once a week but no more than once a day (For use of non-dedicated equipment)	No target compounds > ½ PQL	Investigate source of contamination.	BMcD Chemist	Sensitivity	No target compounds > ½ PQL
Field Duplicate	10% of field samples	See Table 12-2	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Precision	See Table 12-2

Matrix	Water
Analytical Group	Anions (Chloride, Nitrate, Nitrite, Sulfate)
Concentration Level	Low
Sampling SOP	Groundwater FSP Section 6.2
Analytical Method / SOP Reference	EPA Method 300.0
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Table 5-6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
QA Split Sample	10% of field samples	See Table 12-2	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Comparability Precision	See Table 12-2
Not Applicable	Each Sample	Sample analyzed with the applicable method holding time	Reject Data	BMcD Chemist	Representativeness	Sample analyzed with the applicable method holding time

QAPP Worksheet #28-11 Sulfide analysis using EPA 376.1 (Water)

Matrix	Water
Analytical Group	Sulfide
Concentration Level	Low
Sampling SOP	Groundwater FSP Section 6.2
Analytical Method / SOP Reference	EPA Method 376.1
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Table 5-6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per Batch (20 samples)	No target compounds > ½ PQL	See I-Sulfide-Titrimetry-RC	Analyst	Sensitivity Accuracy/Bias – Contamination	No target compounds > ½ PQL
LCS	1 per Batch (20 samples)	See Table 12-10	See I-Sulfide-Titrimetry-RC	Analyst	Accuracy/Bias	REC within limits
MS/MSD Samples	5% of field samples (1 site-specific per Batch)	See Table 12-10	See I-Sulfide-Titrimetry-RC	Analyst	Accuracy/Bias/ Precision	REC within limits RPD within limits
Laboratory Duplicate	1 per Batch (20 samples)	RPD ≤ 25% for each target compound	Perform second duplicate analysis. If the results are still unacceptable and the associated samples are not reanalyzed, flag the data.	Analyst	Precision	RPD ≤ 25% for each target compound
Rinsate Blank	20% of field samples; Once a week but no more than once a day (For use of non-dedicated equipment)	No target compounds > ½ PQL	Investigate source of contamination.	BMcD Chemist	Sensitivity	No target compounds > ½ PQL
Field Duplicate	10% of field samples	See Table 12-2	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Precision	See Table 12-2

Matrix	Water
Analytical Group	Sulfide
Concentration Level	Low
Sampling SOP	Groundwater FSP Section 6.2
Analytical Method / SOP Reference	EPA Method 376.1
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Table 5-6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
QA Split Sample	10% of field samples	See Table 12-2	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Comparability Precision	See Table 12-2
Not Applicable	Each Sample	Sample analyzed with the applicable method holding time	Reject Data	BMcD Chemist	Representativeness	Sample analyzed with the applicable method holding time

QAPP Worksheet #28-12 TOC analysis using SW-846 9060 (Water)

Matrix	Water
Analytical Group	TOC
Concentration Level	Low
Sampling SOP	Groundwater FSP Section 6.2
Analytical Method / SOP Reference	SW-846 9060
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Table 5-6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per Batch (20 samples)	No target compounds > ½ PQL	See I-TOC-RC	Analyst	Sensitivity Accuracy/Bias – Contamination	No target compounds > ½ PQL
LCS	1 per Batch (20 samples)	See Table 12-10	See I-TOC-RC	Analyst	Accuracy/Bias	REC within limits
MS/MSD Samples	5% of field samples (1 site-specific per Batch)	See Table 12-10	See I-TOC-RC	Analyst	Accuracy/Bias/ Precision	REC within limits RPD within limits
Laboratory Duplicate	1 per Batch (20 samples)	RPD ≤ 25% for each target compound	Perform second duplicate analysis. If the results are still unacceptable and the associated samples are not reanalyzed, flag the data.	Analyst	Precision	RPD ≤ 25% for each target compound
Rinsate Blank	20% of field samples; Once a week but no more than once a day (For use of non-dedicated equipment)	No target compounds > ½ PQL	Investigate source of contamination.	BMcD Chemist	Sensitivity	No target compounds > ½ PQL
Field Duplicate	10% of field samples	See Table 12-2	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Precision	See Table 12-2

Matrix	Water
Analytical Group	TOC
Concentration Level	Low
Sampling SOP	Groundwater FSP Section 6.2
Analytical Method / SOP Reference	SW-846 9060
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Table 5-6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
QA Split Sample	10% of field samples	See Table 12-2	Evaluation of source of deviance. Potential resampling.	BMcD Chemist	Comparability Precision	See Table 12-2
Not Applicable	Each Sample	Sample analyzed with the applicable method holding time	Reject Data	BMcD Chemist	Representativeness	Sample analyzed with the applicable method holding time

QAPP Worksheet #28-13 On-site VOC analysis by field GC (Water)

Matrix	Water
Analytical Group	VOCs
Concentration Level	Low
Sampling SOP	Direct-Push Groundwater FSP Section 6.2
Analytical Method / SOP Reference	See *FPM* (EPS) Appendix B
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Tables 5-2 through 5-6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Reporting Limit Verification	Prior to start of field activities or after system maintenance	See *FPM* (Appendix B)	See Field Procedures Manual	EPS	Accuracy/Bias	See *FPM* (Appendix B)
Initial Calibration Standards	Prior to start of field activities or after system maintenance	See *FPM* (Appendix B)	See Field Procedures Manual	EPS	Accuracy/Bias	See *FPM* (Appendix B)
Continuing Calibration Standard	Beginning and end of each day of analysis. Also, 1 per 10 samples.	See *FPM* (Appendix B)	See Field Procedures Manual	EPS	Accuracy/Bias	See *FPM* (Appendix B)
Method Blank	Beginning of each day and following every 10th sample	See *FPM* (Appendix B)	See Field Procedures Manual	EPS	Sensitivity Accuracy/Bias – Contamination	See *FPM* (Appendix B)
Rinsate Blank	Collected prior to sampling from field equipment	See *FPM* (Appendix B)	See Field Procedures Manual	EPS	Sensitivity Accuracy/Bias – Contamination	See *FPM* (Appendix B)
Duplicates	10% of field samples	See *FPM* (Appendix B)	See Field Procedures Manual	EPS	Precision	See *FPM* (Appendix B)

Matrix	Water
Analytical Group	VOCs
Concentration Level	Low
Sampling SOP	Direct-Push Groundwater FSP Section 6.2
Analytical Method / SOP Reference	See *FPM* (EPS) Appendix B
Sampler's Name	TBD
Field Sampling Organization	BMcD
Analytical Organization	AML
Number of Sample Locations	See FSP Tables 5-2 through 5-6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Off-Site Confirmation	10% of field GC samples	Linear Regression (r^2) ≥ 0.70	See Worksheets #12 and #28	See Worksheets #12 and #28	Precision	Linear Regression Off-Site vs Field GC (r^2 ≥ 0.70)
Confirmation samples will be collected at a frequency of 10 percent and submitted to an off-site lab for analysis of VOCs. Guidelines for on-site analysis is provided in Appendix B and documentation guidelines are presented in Appendix E.						

QAPP Worksheet #29
(UFP-QAPP Manual Section 3.5.1)
Project Documents and Records Table

Project Reports and Deliverables are described in additional detail in WP Section 6.0. FSP Section 7.0 presents information for field documentation.

Sample Collection Documents and Records	On-Site Analysis Documents and Records	Off-Site Analysis Documents and Records	Data Assessment Documents and Records	Other
Field Notes, Field Forms, and Photographic Records	GC Initial Standardization Info	Sample receipt and log-in information	DQCR	RI Report Addendum
COC Records	Raw Chromatograms	Analytical hard copy data packages	Monthly Progress Report	BLRA
Groundwater Sampling Report	Notebook documentation for screening samples	SEDD 2a electronic deliverable	QCSR	FS Report
Boring Logs	On-site Data Report	Copies of instrument calibration records, maintenance records, raw data, etc. should be maintained as indicated in lab *QAP*.	Telephone and Email Logs	
Air Bills	Boring Logs			
Custody Seals				
Telephone and Email Logs				

DATA PACKAGE DELIVERABLES

Sample Collection and Field Measurements Data Package Deliverables

Field measurements will be collected using a PID during all field activities. Information pertaining to equipment maintenance, calibration, and calibration verification of the PID will be maintained in the field logbook. Field measurements of temperature, pH, specific conductance, dissolved oxygen, oxidation reduction potential, and turbidity will be collected during purging of each well and prior to collection of each groundwater sample. A ferrous iron measurement will also be collected for evaluation as a natural attenuation indicator. Each of these measurements will be recorded in the field logbook and on the Groundwater Sampling Report. (See FSP Section 7.2).

On-Site Analytical Data Package Deliverables

Groundwater samples collected during direct-push activities will be analyzed by the on-site laboratory using a field GC (EPS). EPS will provide analytical results for each field sample, blank sample, and standard analyzed. The initial calibration and reporting limit verification results will also be provided. EPS will document their activities as indicated in *Guidelines for Analysis Notebook Documentation for Screening Samples* in Appendix E.

Off-Site Laboratory Data Package Deliverables

AML is providing the analytical services for the soil, sediment, and water samples (see Worksheet #30). AML will provide analytical results for each sample collected during the investigation within 21 days of receipt of the last sample for each event.

Laboratory data packages deliverables from AML will include the following information:

- Case narrative – A case narrative will be provided by the lab to include basic project information and a summary of any analytical problems and their resolution.
- Shipping/Receiving Documents – Shipping/Receiving documents provided by the lab will include airbills, COC forms, and sample receipt or log-in sheets.

- Sample Data/Results – Sample data/results will include the field sample name, its associate lab identification number, method name, results for each target analyte with appropriate units, data qualifiers, MDLs and PQLs for all compounds, and identification of analytical batch.

- QC Summary Data - QC summary data will be provided for all method blanks, laboratory duplicates, surrogate spikes, LCS, and/or MS/MSD samples. This information will include identification of the analytical batch, its association with the field sample, results for the QC sample, acceptance criteria for the QC sample, and identification of any corrective actions that were required.

Additional information regarding AML's analytical reports is located in their *QAP* (See Appendix A).

DATA REPORTING FORMATS

Data reporting guidelines for field forms and logbooks is provided in FSP Section 7.2. Copies of field forms for the investigation are provided in FSP Appendix B.

Analytical data flow will be as follows:

- Data will be provided by AML as both hardcopy and electronic submittal (SEDD 2a format; see below)
- Data structure will be verified using automated data review (ADR), and after passing verification, be validated by the BMcD Chemist
- Qualifiers may be added using whatever interface is most efficient for the BMcD Chemist, resulting in updating the SEDD with appropriate qualifiers
- SEDD will again be verified using ADR
- SEDD will be appended to the existing data (Environmental Data Management System [EDMS] format)
- Data will be submitted to CENWK on a quarterly basis (March, June, September, December). Each submittal will include the entire EDMS database as of the 30th of the month and any validated SEDDs that were appended since the last delivery. Only data updated through the 15th of the submittal month will be included
- Delivery will be made on CD or DVD, but upon request, may be uploaded to the CENWK-FTP server when expedience is required

As noted, the electronic submittal of the analytical data will be provided in SEDD 2a format (See www.epa.gov/superfund/programs/clp/sedd.htm). Data reporting guidelines and relevant forms and checklists for AML are provided in their *QAP* and SOPs. (See Appendix A)

DATA HANDLING AND MANAGEMENT

Data recording, transformation, reduction, transfer, and analysis information for AML are provided in their *QAP* and SOPs. (See Appendix A)

DATA TRACKING AND CONTROL

The BMcD Project Manager has the overall responsibility for data tracking and control. These data management activities include record-keeping, tracking, document control systems, and data handling to process, compile, analyze, and transmit data. Day-to-day oversight of sampling activities, laboratory activities, and data tracking and receipt will be the responsibility of the BMcD Project Manager or a designated project team member.

All project teams members are responsible for handling data in a manner consistent with procedures listed in Sections 7.0 (Field Operations Documentation) and 8.0 (Sample Packaging and Shipping Requirements) and their associated appendices. These sections include information pertaining to field logbooks, sample numbering, sample documentation, laboratory assignments, documentation (cooler/shipping documentation and filing system), and corrections to documentation.

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QAPP Worksheet #30
(UFP-QAPP Manual Section 3.5.2.3)
Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Locations/ ID Number	Analytical SOP	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	QA Split Sample Laboratory / Organization (name and address, contact person and telephone number)
Soil	VOCs	Low	See FSP Tables 5-2 through 5-5 Figures 5-1 through 5-8	SW-846 8260B	21 Days	Analytical Management Laboratories, Inc. 15130 South Keeler Street Olathe, Kansas 66062 Phone: (913) 829-0101 Contact: Dr. Tenkasi S. Viswanathan	ECB Laboratory 3909 Halls Ferry Road Vicksburg, Mississippi 39180 Phone: (601) 634-3652 Contact: Dr. Tony Bednar
	SVOCs			SW-846 8270D			
	Metals			SW-846 6020A			
	Mercury			SW-846 7471A			
Water	VOCs	Low	See FSP Tables 5-2 through 5-6 Figures 5-1 through 5-8	SW-846 8260B	21 Days	Analytical Management Laboratories, Inc. 15130 South Keeler Street Olathe, Kansas 66062 Phone: (913) 829-0101 Contact: Dr. Tenkasi S. Viswanathan	ECB Laboratory 3909 Halls Ferry Road Vicksburg, Mississippi 39180 Phone: (601) 634-3652 Contact: Dr. Tony Bednar
	SVOCs			SW-846 8270D			
	MEE			RSK-175			
	Metals			SW-846 6020A			
	Mercury			SW-846 7470A			
	Anions			EPA 300.0			
	TOC			SW-846 9060			
	Sulfide			EPA 376.1			
Copies of AML's SOPs for each analytical method are provided in Appendix A.							

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QAPP Worksheet #31
(UFP-QAPP Manual Section 4.1.1)
Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organizational affiliation)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
DQCR	Daily during field activities	Internal	BMcD	FSM	Ed Lindgren, BMcD, Technical Lead	FSM and BMcD Technical Lead	BMcD Technical Lead
Confirmation Samples	Confirmation samples at 10% of direct-push groundwater samples	External	BMcD	Shauna Lawrence, BMcD, Chemist	Ed Lindgren, BMcD, Technical Lead	Ed Lindgren, BMcD, Technical Lead	Shauna Lawrence, BMcD, Chemist
Field Duplicate Samples	Field duplicate samples at 10% of soil samples and 10% of water samples	External	USACE	Shauna Lawrence, BMcD, Chemist	Ed Lindgren, BMcD, Technical Lead	Ed Lindgren, BMcD, Technical Lead	Shauna Lawrence, BMcD, Chemist
QA Split Samples	QA Split samples at 10% of soil samples and 10% of water samples	External	USACE	ECB Laboratory	Ed Lindgren, BMcD, Technical Lead	Ed Lindgren, BMcD, Technical Lead	Shauna Lawrence, BMcD, Chemist
PE Sample	One PE sample for VOCs (Groundwater)	External	BMcD	Shauna Lawrence, BMcD, Chemist	Ed Lindgren, BMcD, Technical Lead	Ed Lindgren, BMcD, Technical Lead	Shauna Lawrence, BMcD, Chemist

CONFIRMATION SAMPLING, SPLIT SAMPLING, AND ANALYSIS AUDIT

Confirmation samples are planned for 10 percent of the groundwater samples analyzed on-site using a field GC during the direct-push field activities. These confirmation samples will be of groundwater matrix and analyzed off-site by AML for VOC confirmation analysis. Field duplicates and QA split samples are planned for 10 percent of the samples sent to an off-site lab for analysis. The QA split samples will be analyzed by ECB Laboratory. These samples will be of groundwater, surface water, sediment, and soil matrices. Comparisons of the data between the on-site GC/off-site confirmation and original/field duplicate/QA samples will be performed following the criteria outlined in Worksheet #12.

FIELD PERFORMANCE AND SYSTEM AUDIT

One or more field audits will be performed during the course of the OU 1 investigation. Field audits are subject to inspections by one or more of the following project team members: QC Coordinator, Project Geologist, Project Chemist, Health and Safety Personnel, and any other applicable project team members. Findings of the field audit will be reported to the Project Managers (BMcD and MP) and reported in the applicable reports. Notes of who, when, why, etc. will be noted in the field book and appropriate field records.

PERFORMANCE EVALUATION SAMPLES

One blind PE sample for VOCs in groundwater will be submitted to the primary lab (AML) for analysis by SW-846 8260B to provide information regarding the accuracy and capabilities of the lab. The PE sample provides a measure of laboratory performance that is used along with other QA criteria to monitor laboratory capability. This PE sample will be submitted to AML as a blind sample along with other field samples collected during the sampling activities. The PE sample will be provided by Wibby Environmental. Wibby Environmental will provide acceptable control limits for the PE sample to BMcD for comparison to the laboratory results reported by AML by criteria established in Worksheet #12. Results of the PE sample by AML will be discussed in the QCSR.

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QAPP Worksheet #32
(UFP-QAPP Manual Section 4.1.2)
Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
DQCRs	Notification of FSM, Weekly Progress Report	Robyn Kiefer, Project Manager, CENWK Tracy Cooley, Project Manager, BMcD John Logigian, Project Manager, MP	ASAP	Notify field crew for corrective actions (i.e., resampling) while still on site	Ed Lindgren, Technical Lead BMcD	ASAP
PE sample	Written Data Comparison Memo	Robyn Kiefer, Project Manager, CENWK Tracy Cooley, Project Manager, BMcD John Logigian, Project Manager, MP	One week following receipt of data	Letter	Shauna Lawrence, Project Chemist, BMcD	One week following receipt of letter
Split Sampling, Analysis Audit	Chemical Quality Assurance Report (CQAR)	Robyn Kiefer, PM, CENWK	Following analyst review of data by QA lab	Letter	Shauna Lawrence, Project Chemist, BMcD	One week following receipt of letter

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Confirmation Samples	Written Data Comparison Memo	Robyn Kiefer, Project Manager, CENWK Tracy Cooley, Project Manager, BMcD John Logigian, Project Manager, MP	One week following receipt of data	Letter	Shauna Lawrence, Project Chemist, BMcD	One week following receipt of letter

ASSESSMENT FINDINGS AND CORRECTIVE ACTION RESPONSES

The findings of any assessments or audits will be provided in a brief report to the BMcD Project Manager. This brief report will contain the results of the evaluation and recommendations for corrective actions. The BMcD QC Coordinator will be responsible for ensuring that an appropriate response is made regarding the audit findings and corrective actions are implemented.

Corrective actions will be taken when deficiencies are identified. Corrective actions will be determined based on the specific deficiency and the impact of the deficiency on the technical quality of the project. Since some deficiencies are more serious than others, each deficiency will be reviewed to determine the corrective action that is most appropriate. Factors to be considered in this evaluation include the cause of the deficiency and the impact of the deficiency on project goals. An assessment will be made regarding any effect of the deficiency on project budgets, schedules, and technical adequacy; and the appropriate corrective action will be determined.

Deficiencies detected during execution of the project will be fully explained to the project team by the BMcD Project Manager, either at the time the deficiency is noted or at the scheduled project team meetings. If possible, deficiencies will be corrected "on-the-spot" in accordance with QC personnel direction. Deficiencies related to health and safety will be corrected immediately to protect worker health and safety. For deficiencies that have a significant

impact on the project, an appropriate corrective action will be determined and implemented by the BMcD Project Manager following consultation with the MP and CENWK Project Managers. QC personnel will document deficiencies and how they are corrected. The Project Manager will be responsible for informing CENWK and MP of deficiencies through external reporting and direct conversations.

The results of the PE sample will be compared to the results reported by the primary lab (AML). The PE sample is a sample of known concentration that is analyzed by the lab and the results are compared with the certified concentration(s). The PE sample provides a measure of laboratory performance that is used along with other QA criteria to monitor laboratory capability. The Project Chemist will evaluate the analytical results and report discrepancies to the BMcD Project Manager as soon as possible after discovering the problem. Further actions will be determined and implemented by the BMcD Project Manager following consultation with the MP and CENWK Project Managers and/or Chemists.

In the field, the FSM will be responsible for observing field activities and checking the field project team members' work for completeness, consistency, and accuracy. If a deficiency is noted, the FSM will be responsible for implementing "on-the-spot" corrections, as appropriate, to address the deficiency and to minimize future occurrence of a similar deficiency. If a deficiency cannot be easily corrected "on-the-spot", the FSM will immediately notify the BMcD Project Manager so the situation can be fully addressed and resolved. In the office, Project Team members will be responsible for checking their work products prior to submission to the BMcD Project Manager for review. The BMcD Project Manager will be responsible for reviewing completed work products and providing Project Team members with comments, corrections, and revisions.

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QAPP Worksheet #33
(UFP QAPP Manual Section 4.2)
QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
DQCR	Daily	Daily during field sampling activities	FSM, BMcD	Tracy Cooley, PM, BMcD John Logigian, PM, MP Robyn Kiefer, PM, CENWK
Monthly Progress Report	Monthly	15th of each month	Ed Lindgren, Technical Lead, BMcD John Logigian, PM, MP	Robyn Kiefer, PM, CENWK
Quality Control Summary Report	Once – after receipt of analytical data (See WP Section 5.3)	See Worksheet #16	Tracy Cooley, PM, BMcD Shauna Lawrence, Project Chemist, BMcD	Robyn Kiefer, PM, CENWK Jorge Jacobs, KDHE Ken Rapplean, USEPA Restoration Advisory Board
Remedial Investigation Report Addendum	Once – after completion of the RI (See WP Section 5.7)	See Worksheet #16	John Logigian, PM, MP Tracy Cooley, Project Manager, BMcD	Robyn Kiefer, PM, CENWK Jorge Jacobs, KDHE Ken Rapplean, USEPA Restoration Advisory Board
Feasibility Study Report	Once – after completion of the FS (See WP Section 5.10)	See Worksheet #16	John Logigian, PM, MP Tracy Cooley, Project Manager, BMcD	Robyn Kiefer, PM, CENWK Jorge Jacobs, KDHE Ken Rapplean, USEPA Restoration Advisory Board
Proposed Plan	Once	See Worksheet #16	John Logigian, PM, MP Tracy Cooley, Project Manager, BMcD	Robyn Kiefer, PM, CENWK Jorge Jacobs, KDHE Ken Rapplean, USEPA Restoration Advisory Board
Record of Decision	Once	See Worksheet #16	John Logigian, PM, MP Tracy Cooley, Project Manager, BMcD	Robyn Kiefer, PM, CENWK Jorge Jacobs, KDHE Ken Rapplean, USEPA Restoration Advisory Board

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Interim Record of Decision	Once	See Worksheet #16	John Logigian, PM, MP Tracy Cooley, Project Manager, BMcD	Robyn Kiefer, PM, CENWK Jorge Jacobs, KDHE Ken Rapplean, USEPA Restoration Advisory Board
See WP Section 5.0 for further details on each report.				

QA MANAGEMENT REPORTS

Periodic QA management reports are planned to update project team members on project status and the result of QA assessments. The following QA Management Reports are being developed for the project (See Worksheet #31):

- DQCRs
- Monthly Progress Reports
- QCSR

FINAL PROJECT REPORT

A RI Report Addendum will be submitted as indicated in WP Section 5.7. This report will be prepared to present all pertinent data to the investigation. A FS Report will be submitted following completion of the RI Report Addendum as indicated in WP Section 5.10. This report will include a discussion of the development of remedial alternatives. Data usability for samples collected for these reports will be presented in a QCSR as noted above.

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QAPP Worksheet #34
(UFP-QAPP Manual Section 5.2.1)
Verification (Step I) Process Table

This worksheet and text describes the processes that will be followed to verify project data. *Internal* or *external* is in relation to the data generator.

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
COC and Shipping Forms	COC and shipping forms will be reviewed by the FSM upon their completion. The documents will be verified against the sample shipment. See FSP Sections 7.4.3 and 8.0 for more information.	I	FSM, BMcD
DQCR	DQCRs will be reviewed by the QC Coordinator for completeness and correctness prior to submittal to CENWK and MP. See FSP Section 7.1 for more information.	I	Tracy Cooley, Project Manager, BMcD
Field Notes, Field Forms, and Photographic Records	Field Notes and Photographic Records will be reviewed internally and placed in the project file. See FSP Sections 7.2 and 7.3 for more information.	I	Ed Lindgren, Technical Lead, BMcD
Laboratory Data	Lab data packages will be reviewed by AML internally for completeness and accuracy prior to submittal.	I	QA Director, AML
	Data packages will be reviewed externally according to data validation procedures established in the QAPP (See Worksheets #35 and #36)	E	Shauna Lawrence, Project Chemist, BMcD

Step I – Verification

Data Review Step I is called Verification. Verification includes a review for completeness and that all data required for the project is available.

The laboratory performs verification of data prior to its release to the customer. AML's data verification procedures are described in Section 12 of their *QAP* (See Appendix A) and follows a three-level approach for data verification as indicated in DoD QSM Box 42.

Verification requires knowledge of the type of information that is reviewed. Therefore, a person familiar with field activities, such as the FSM or site geologist, is typically assigned to the review of field activities, documents, and records. Likewise, a person familiar with analytical methodology, such as a chemist, is typically assigned to the review of laboratory documents and records.

BMcD evaluates data quality through the evaluation of both field and laboratory QC data. Verification is initiated at the time of first sample collection. Field documents are reviewed by the FSM or a designee to determine that all samples and analyses were appropriately collected, containerized, labeled, and submitted to the laboratory. These items will be verified daily during sampling activities. Additionally, the project chemist or designee will be in communication with the lab during sample collection and analysis to verify condition of sample receipt, appropriate sample log-in, etc. If problems are noted at this point, they can easily be corrected or locations resampled, if needed, while the field crews are still mobilized.

* * * * *

QAPP Worksheet #35
(UFP-QAPP Manual Section 5.2.2)
Validation (Steps IIa and IIb) Process Table

This worksheet describes the processes that will be followed to validate project data. Validation inputs include items such as those listed in Table 9 of USP-QAPP Manual (Section 5.1). It describes how each item will be validated, when the activity will occur, what documentation is necessary, and the identity of the person responsible. It also differentiates between the Steps IIa and IIb of validation.

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	SOPs	Ensure that sampling and analytical SOPs were followed.	Shauna Lawrence, Project Chemist, BMcD
IIa	Analytical QC Sample Data	Verify that all method-required QC samples were analyzed, and results met the method requirements.	Shauna Lawrence, Project Chemist, BMcD
IIb	Field QC Sample Data	Verify that all QAPP-required QC samples were analyzed, and results met the QAPP requirements.	Shauna Lawrence, Project Chemist, BMcD
IIb	PQLs	Verify that the sample results met the project PQLs specified in the QAPP.	Shauna Lawrence, Project Chemist, BMcD

* * * * *

QAPP Worksheet #36
(UFP-QAPP Manual Section 5.2.2)
Validation (Steps IIa and IIb) Summary Table

This worksheet and text identifies the matrices, analytical groups, and concentration levels that each entity performing validation will be responsible for, as well as criteria that will be used to validate those data.

Step IIa / IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa / IIb	Soil/Water	VOCs	Low	CENWK *Data Quality Evaluation Guidance* (see QAPP Appendix D)	Shauna Lawrence, Project Chemist, BMcD
IIa / IIb	Soil/Water	SVOCs			
IIa / IIb	Soil/Water	Metals			
IIa / IIb	Water	MEE			
IIa / IIb	Water	Anions			
IIa / IIb	Water	Sulfide			
IIa / IIb	Water	TOC			
The majority of the validation guidance comes from the CENWK Data Quality Evaluation Guidance (Appendix D). Specific details relevant to validation IIa activities can be found in the specific analytical method noted above, while specific details to relevant validation IIb activities can be found in QAPP Worksheets #12, 15, 20, 23, 24, 25, and 28.					

STEP II – VALIDATION

Validation includes evaluating compliance with method, procedure, or contract requirements and extends into evaluating the data against MPC that were established in the QAPP. The results of data validation will be documented in the QCSR that will be submitted to discuss the quality of the soil, sediment, surface water, and groundwater data collected and analyzed during the investigation.

QAPP Worksheet #12 provided a discussion of the DQIs (precision, accuracy/bias, sensitivity, representativeness, comparability, and completeness) that will be evaluated as part of validation. The quality of the data will be assessed through evaluation of the results of the field QC samples (trip blanks, field

duplicates, confirmation samples, PE sample, and QA split samples) and laboratory internal QC samples (method blanks, surrogates, lab duplicates, and LCSs). Data qualifiers, when appropriate, will be added to the data in accordance with the *Kansas City District Data Quality Evaluation Guidance* (USACE, 2006), which is provided in Appendix D. A brief summary is presented in the following paragraphs:

- Precision – Precision for the field duplicate samples will be evaluated by criteria presented in Tables 12-1 (soil/sediment) and Table 12-2 (water). Precision for the confirmation samples will be by linear regression (See Worksheet #12). A CQAR will be submitted to CENWK for precision comparison of the QA split samples sent to ECB Laboratory. For all other duplicate analyses (i.e. laboratory duplicates, MS/MSD pairs, etc.,) the RPD will be calculated as presented in Worksheets #12 and #28 for the respective analysis. Precision criteria outside of QC limits may result in qualification of data as estimated (J).

- Accuracy/Bias – Accuracy will be assessed by evaluating the results of spiked samples for REC and blank samples for potential contamination of samples. REC results for spike samples (surrogates and LCS) will be used to assign qualifiers to analytical data. A REC above QC limits suggests the possibility of high bias in the analytical results. A REC below QC limits suggests the possibility of low biased or false negative analytical results. Data will be qualified as estimated (J) or rejected (R) based upon the magnitude of the deviance from QC limits.

  Accuracy for the PE sample will be assessed by comparing the certified acceptable control limits provided by Wibby Environmental compared to the results submitted by AML. Data will be qualified as estimated (J) or rejected (R) based upon the magnitude of the deviance from QC limits.

  Blank samples will be used to evaluate whether field samples have been cross-contaminated during shipping or handling. Detections in blank samples indicate the potential for high-biased or false positive data. Data will be qualified as undetected (U) or J based upon the magnitude of the blank detection relative to the result in the field sample.

- Representativeness – Representativeness will be assessed by examining sample preservation, results of the precision and accuracy evaluation, and adherence to method holding time. Failure of field or laboratory personnel to properly handle samples may result in qualification of the data as estimated or unusable. The representativeness review will qualitatively consider whether precision and/or accuracy are sufficient to characterize the samples. Analytical data for samples that are not analyzed within holding times will be qualified R as indicated in Appendix D.

- Completeness – Completeness will be assessed by calculation of field completeness and laboratory completeness as presented on Worksheet #12.

- Comparability – Comparability will be assessed by evaluating QA split sample data, confirmation sample data, and/or PE sample data as presented on Worksheet #12.

Step IIa Validation Activities

Step IIa validation includes evaluation of the data with MPC that were established for the applicable method and SOP. Information regarding MPC are presented on Worksheets #12, #15, #20, #23, #24, #25, and #28. Following receipt of the analytical data packages, the Step IIa validation will include review of the following items:

- Sample analysis occurred within holding time
- Method blank results
- Laboratory Duplicates
- Surrogate REC
- LCS REC
- MS/MSD REC/RPD

Data qualifiers, when appropriate, will be added to the data in accordance with recommendations in the *Kansas City District Data Quality Evaluation Guidance* (USACE-CENWK, 2006), which is provided in Appendix D.

Step IIb Validation Activities

Step IIb validation includes evaluation of the data with MPC that were established in the QAPP. Information regarding MPC is presented on Worksheets #12, #15, #20, #23, #24, #25, and #28 in Appendix A. Following receipt of the analytical data packages, the Step IIb validation will include review of the following items:

- Requested analyses performed using QAPP-specified method
- Trip Blanks
- Rinsate Blanks
- Confirmation sample results
- Comparison to PE sample results
- Field Duplicates
- Achievement of project-required quantitation PQLs
- Field and Laboratory Completeness
- Comparability of QA Split Samples

Data qualifiers, when appropriate, will be added to the data in accordance with the *Kansas City District Data Quality Evaluation Guidance* (CENWK, 2006), which is provided in Appendix D.

QAPP Worksheet #37
(UFP-QAPP Manual Section 5.2.3)
Usability Assessment

Data review extends beyond method, procedural, and contractual compliance to determine the quality of the data set and the types of uncertainty introduced by a failure to meet requirements. It includes a determination, where possible, of the reasons for any failure to meet requirements, and an evaluation of the impact of the failure upon the overall data set. In this manner, the effect of any data rejection is presented in terms of its impacts on the overall uncertainty and usability of the data set.

DQIs of precision, accuracy/bias, sensitivity, representativeness, comparability, and completeness provide important information regarding the overall usability of the data. If DQIs do not meet the requirements as outlined (See QAPP Worksheet #12) or problems are noted with sample collection, the data may be discarded and re-sampling may occur. The BMcD Project Manager will make this decision after consultation with the other key project personnel.

Summarize the usability assessment process and all procedures, including interim steps any statistics, equations, and computer algorithms that will be used:

Steps utilized for assessing data usability are based on guidance provided in "*Guidance for Evaluating Performance-Based Chemical Data* (USACE, 2005). Data must be assessed for overall usability on the basis of total measurement uncertainty as well as the objectives of the investigation. This total measurement uncertainty consists of both laboratory analytical uncertainty and field sampling uncertainty, with the latter having greater uncertainty because it is not fully taken into account during data review or validation.

See QAPP Worksheet #12 for discussion of calculation/evaluation of data quality indicators of precision, accuracy/bias, sensitivity, comparability, completeness, and representativeness.

Usability assessment is typically performed after data review or data validation. Data quality is evaluated during data review in the context of the end use of the data.

Describe the evaluative procedures used to assess overall measurement error associated with the project:

After data validation activities have been completed, the data usability assessment will be performed by a team of BMcD personnel, including the Project Manager, Project Chemist, and QC Coordinator. This

team will perform a global review of the findings to determine overall usability of the data set for its intended purpose. It is at this point that a final analysis of the data is made, taking into consideration the following:

- Sample collection – Were problems encountered during sample collection that suggest samples were potentially compromised? If so, what is the impact?
- Suitability of methodology - Based upon the chemical data validation, were significant precision or bias problems noted with the data? Were significant matrix interference problems noted?
- Adequacy of reporting limits - Was excessive sample dilution required due to interference or presence of elevated concentrations of target or nontarget compounds? If so, does this adversely impact the ability to draw conclusions regarding any undetected constituents?
- Reasonableness of QC limits – Do the initially established control limits for DQIs still seem appropriate for the data set? If not, is the data exhibiting higher variability than assumed during project planning?
- Patterns in qualified data – Are patterns evident in the type of samples or analyses that required qualification during validation? Do these patterns suggest overall problems in one area or for a particular type of analysis?

Identify the personnel responsible for performing the usability assessment:

The data usability assessment will be performed by a team of personnel at BMcD. Tracy Cooley, Project Manager, will oversee the usability assessment. Supporting team members include: Shauna Lawrence, Project Chemist and Ed Lindgren, Technical Lead.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:

A QCSR will be generated to present the results of the data validation performed according to Worksheet #35 and Worksheet #36. The results of the usability assessment will be documented in a section of the RI Report Addendum and the FS Report.

* * * * *

TABLES

Table 11-1 – Analyte List

Table 12-1 – Field Duplicate Sample Comparison for Soil/Sediment Samples

Table 12-2 – Field Duplicate Sample Comparison for Water Samples

Tables 12-3 through 12-12 QC Sample Acceptance Limits

Table 12-13 Cross Reference for Laboratory Reporting Limits

Tables 24-1 through 24-8 Summary of Calibration and Quality Control Procedures

Table 11-1
Analyte List
RI Addendum/FS
Former Schilling Air Force Base

VOCs by SW-846 8260B		
Dichlorodifluoromethane	Trichloroethene	1,1,2,2-Tetrachloroethane
Chloromethane	1,2-Dichloropropane	Bromobenzene
Vinyl Chloride	Dibromomethane	n-Propylbenzene
Bromomethane	Bromodichloromethane	2-Chlorotoluene
Chloroethane	cis-1,3-Dichloropropene	4-Chlorotoluene
1,1-Dichloroethene	4-Methyl-2-pentanone	1,3,5-Trimethylbenzene
Acetone	Toluene	tert-Butylbenzene
Carbon Disulfide	trans-1,3-Dichloropropene	1,2,4-Trimethylbenzene
Methylene Chloride	1,1,2-Trichloroethane	Sec-Butylbenzene
trans-1,2-Dichloroethene	1,3-Dichloropropane	1,3-Dichlorobenzene
Methyl-tert-butyl-ether	Tetrachloroethene	1,4-Dichlorobenzene
1,1-Dichloroethane	2-Hexanone	1,2-Dichlorobenzene
2-Butanone	Dibromochloromethane	4-Isopropyltoluene
cis-1,2-Dichloroethene	1,2-Dibromoethane	n-Butylbenzene
2,2-Dichloropropane	Chlorobenzene	1,2,4-Trichlorobenzene
Bromochloromethane	1,1,1,2-Tetrachloroethane	1,2,3-Trichlorobenzene
Chloroform	Ethylbenzene	Hexachlorobutadiene
1,1,1-Trichloroethane	m-Xylene and p-Xylene	Naphthalene
1,1-Dichloropropene	o-Xylene	1,2,3-Trichloropropane
Carbon Tetrachloride	Styrene	1,2-Dibromo-3-chloropropane
1,2-Dichloroethane	Bromoform	Trichlorofluoromethane
Benzene	Isopropylbenzene	

SVOCs by SW-846 8270D		
bis(2-Chloroethyl)ether	Acenaphthene	Flouranthene
bis(2-chloroisopropyl)ether	Acenaphthylene	Hexachlorobenzene
2-Chlorophenol	2-Chloronaphthalene	Pentachlorophenol
1,3-Dichlorobenzene	4-Chlorophenyl-phenylether	Benzo(a)anthracene
1,4-Dichlorobenzene	Dibenzofuran	Chrysene
1,2-Dichlorobenzene	Diethylphthalate	bis(2-ethylhexyl)phthalate
Hexachloroethane	Dimethylphthalate	Butylbenzylphthalate
2-Methylphenol (o-Cresol)	2,4-Dinitrophenol	Pyrene
¾-Methylphenol (m/p-Cresol)	2,6-Dinitrotoluene	Di-n-octylphthalate
N-Nitroso-di-n-propylamine	2,4-Dinitrotoluene	Indeno(1,2,3-cd)pyrene
Phenol	Fluorene	Benzo(b)fluoranthene
bis(2-chloroethoxy)methane	Hexachlorocyclopentadiene	Benzo(k)fluoranthene
4-Chloroaniline	2-Nitroaniline	Benzo(g,h,i)perylene
4-Chloro-3-methylphenol	3-Nitroaniline	Benzo(a)pyrene
2,4-Dichlorophenol	4-Nitroaniline	Dibenz(a,h)anthracene
2,4Dimethylphenol	4-Nitrophenol	Carbazole
Hexachlorobutadiene	2,4,6-Trichlorophenol	Benzoic Acid
Isophorone	2,4,5-Trichlorophenol	3,3'-Dichlorobenzidine
2-Methylnaphthalene	Anthracene	N-Nitrosodimethylamine
Naphthanlene	Phenanthrene	N-Nitrosodiphenylamine
Nitrobenzene	4-Bromophenyl-phenylether	1,2-Diphenylhydrazine
2-Nitrophenol	Di-n-butylphthalate	Benzyl Alcohol
1,2,4-Trichlorobenzene	2-Methyl-4,6-dinitrophenol	

Table 11-1
Analyte List
RI/FS Addendum
Former Schilling Air Force Base

RCRA Metals – SW-846 Methods 6020A and 7470A/7471A (Mercury only)
Arsenic Barium Cadmium Chromium Lead Mercury Selenium Silver

Natural Attenuation Indicator Parameters
MEE by RSK-175
Methane Ethane Ethene
Metals by SW-846 6020A
Iron
Anions by EPA Method 300.0
Chloride Nitrate Nitrite Sulfate
Wet Chemistry
Sulfide by EPA Method 376.1 TOC by SW-846 9060

Note: VOC and SVOC lists were taken from AML's SOPs for the respective analytical method.

EPA = Environmental Protection Agency
MEE = Methane, Ethane, Ethene
RCRA = Resource Conservation Recovery Act
SVOC = Semivolatile Organic Compound
VOC = Volatile Organic Compound

Table 12-1
Field Duplicate Sample Comparison for Soil/Sediment Samples
RI Addendum/FS
Former Schilling Air Force Base

Soil/Sediment Samples	Parameter	Minor Disagreement	Major Disagreement
Criteria for comparing duplicate samples when detections are >PQL in both samples	VOCs	> 5x Difference	> 10x Difference
	Metals	> 2x Difference	> 3x Difference
	All other analyses	> 4x Difference	> 5x Difference
Criteria for comparing duplicate samples when one result is <PQL	All analyses	> 3x Difference	> 5x Difference

PQL = Practical Quantitation Limit
VOCs = Volatile Organic Compounds

Table 12-2
Field Duplicate Sample Comparison for Water Samples
RI Addendum/FS
Former Schilling Air Force Base

Water	Parameter	Minor Disagreement	Major Disagreement
Criteria for comparing duplicate samples when detections are >PQL in both samples	All analyses	> 2x Difference	> 3x Difference
Criteria for comparing duplicate samples when one result is <PQL	All analyses	> 3x Difference	> 5x Difference

PQL = Practical Quantitation Limit

Table 12-3
QC Sample Acceptance Limits
VOCs by SW-846 8260B (Soil/Sediment)
RI Addendum/FS
Former Schilling Air Force Base

Parameter	Control Limits LCS and MS/MSD		ME Control Limits LCS and MS/MSD		RPD Acceptable Limit
	Lower	Upper	Lower	Upper	Limit
	REC		REC		%
Dichlorodifluoromethane	35	135	15	155	30
Chloromethane	50	130	40	140	30
Vinyl Chloride	60	125	45	140	30
Bromomethane	30	160	10	180	30
Chloroethane	40	155	20	175	30
1,1-Dichloroethene	65	135	55	150	30
Acetone	20	160	10	180	30
Carbon Disulfide	45	160	30	180	30
Methylene Chloride	55	140	40	155	30
trans-1,2-Dichloroethene	65	135	55	145	30
Methyl-tert-butyl-ether	65	125	55	135	30
1,1-Dichloroethane	75	125	65	135	30
2-Butanone	30	160	10	180	30
cis-1,2-Dichloroethene	65	125	55	135	30
2,2-Dichloropropane	65	135	55	145	30
Bromochloromethane	70	125	60	135	30
Chloroform	70	125	65	135	30
1,1,1-Trichloroethane	70	135	55	145	30
1,1-Dichloropropene	70	135	60	145	30
Carbon Tetrachloride	65	135	55	145	30
1,2-Dichloroethane	70	135	60	145	30
Benzene	75	125	65	135	30
Trichloroethene	75	125	70	130	30
1,2-Dichloropropane	70	120	65	125	30
Dibromomethane	75	130	65	135	30
Bromodichloromethane	70	130	60	135	30
cis-1,3-Dichloropropene	70	125	65	135	30
4-methyl-2-pentanone	45	145	30	165	30
Toluene	70	125	60	135	30
trans-1,3-Dichloropropene	65	125	55	140	30
1,1,2-Trichloroethane	60	125	50	140	30
1,3-Dichloropropane	75	125	70	130	30
Tetrachloroethene	65	140	55	150	30
2-Hexanone	45	145	30	160	30
Dibromochloromethane	65	130	55	140	30
1,2-Dibromoethane	70	125	60	135	30
Chlorobenzene	75	125	65	130	30
1,1,1,2-Tetrachloroethane	75	125	65	135	30

Table 12-3
QC Sample Acceptance Limits
VOCs by SW-846 8260B (Soil/Sediment)
RI Addendum/FS
Former Schilling Air Force Base

Parameter	Control Limits LCS and MS/MSD		ME Control Limits LCS and MS/MSD		RPD Acceptable Limit
	Lower	Upper	Lower	Upper	Limit
	REC		REC		%
Ethylbenzene	75	125	65	135	30
m-Xylene and p-Xylene	80	125	70	135	30
o-Xylene	75	125	70	135	30
Styrene	75	125	65	135	30
Bromoform	55	135	45	150	30
Isopropylbenzene	75	130	70	140	30
1,1,2,2-Tetrachloroethane	55	130	40	145	30
Bromobenzene	65	120	55	130	30
n-Propylbenzene	65	135	50	145	30
2-Chlorotoluene	70	130	60	140	30
4-Chlorotoluene	75	125	65	135	30
1,3,5-Trimethylbenzene	65	135	55	145	30
tert-Butylbenzene	65	130	55	145	30
1,2,4-Trimethylbenzene	65	135	55	145	30
sec-Butylbenzene	65	130	50	145	30
1,3-Dichlorobenzene	70	125	65	135	30
1,4-Dichlorobenzene	70	125	65	135	30
1,2-Dichlorobenzene	75	120	65	125	30
4-Isopropyltoluene	75	135	65	140	30
n-Butylbenzene	65	140	50	150	30
1,2,4-Trichlorobenzene	65	130	55	140	30
1,2,3-Trichlorobenzene	60	135	50	145	30
Hexachlorobutadiene	55	140	40	155	30
Naphthalene	40	125	25	140	30
1,2,3-Trichloropropane	65	130	50	140	30
1,2-Dibromo-3-chloropropane	40	135	25	150	30
Trichlorofluoromethane	25	185	10	215	30

1) For this VOC analyte list, up to three (3) sporadic MEs are allowed.

AML = Analytical Management Laboratories, Inc.
DoD = Department of Defense
LCS = Laboratory Control Limit
ME = Marginal Exceedence
MS/MSD = Matrix Spike/Matrix Spike Duplicate
QSM = Quality Systems Manual
REC = Percent Recovery
RPD = Relative Percent Difference

Table 12-4
QC Sample Acceptance Limits
VOCs by SW-846 8260B (Water)
RI Addendum/FS
Former Schilling Air Force Base

Parameter	Control Limits LCS and MS/MSD		ME Control Limits LCS and MS/MSD		RPD Acceptable Limit
	Lower	Upper	Lower	Upper	Limit
	REC		REC		%
Dichlorodifluoromethane	30	155	10	175	30
Chloromethane	40	125	25	140	30
Vinyl Chloride	50	145	35	165	30
Bromomethane	30	145	10	165	30
Chloroethane	60	135	50	145	30
1,1-Dichloroethene	70	130	55	140	30
Acetone	40	140	20	160	30
Carbon Disulfide	35	160	15	185	30
Methylene Chloride	55	140	40	155	30
trans-1,2-Dichloroethene	60	140	45	150	30
Methyl-tert-butyl-ether	65	125	55	135	30
1,1-Dichloroethane	70	135	60	145	30
2-Butanone	30	150	10	170	30
cis-1,2-Dichloroethene	70	125	60	135	30
2,2-Dichloropropane	70	135	60	150	30
Bromochloromethane	65	130	55	140	30
Chloroform	65	135	50	150	30
1,1,1-Trichloroethane	65	130	55	145	30
1,1-Dichloropropene	75	130	65	140	30
Carbon Tetrachloride	65	140	55	150	30
1,2-Dichloroethane	70	130	60	140	30
Benzene	80	120	75	130	30
Trichloroethene	70	125	60	135	30
1,2-Dichloropropane	75	125	65	135	30
Dibromomethane	75	125	65	135	30
Bromodichloromethane	75	120	70	130	30
cis-1,3-Dichloropropene	70	130	60	140	30
4-methyl-2-pentanone	60	135	45	145	30
Toluene	75	120	70	130	30
trans-1,3-Dichloropropene	55	140	40	155	30
1,1,2-Trichloroethane	75	125	65	135	30
1,3-Dichloropropane	75	125	65	135	30
Tetrachloroethene	45	150	25	165	30
2-Hexanone	55	130	45	140	30
Dibromochloromethane	60	135	45	145	30
1,2-Dibromoethane	80	120	75	125	30
Chlorobenzene	80	120	75	130	30
1,1,1,2-Tetrachloroethane	80	130	75	135	30

Table 12-4
QC Sample Acceptance Limits
VOCs by SW-846 8260B (Water)
RI Addendum/FS
Former Schilling Air Force Base

Parameter	Control Limits LCS and MS/MSD		ME Control Limits LCS and MS/MSD		RPD Acceptable Limit
	Lower	Upper	Lower	Upper	Limit
	REC		REC		%
Ethylbenzene	75	125	65	135	30
m-Xylene and p-Xylene	75	130	65	135	30
o-Xylene	80	120	75	130	30
Styrene	65	135	55	145	30
Bromoform	70	130	60	140	30
Isopropylbenzene	75	125	65	135	30
1,1,2,2-Tetrachloroethane	65	130	55	140	30
Bromobenzene	75	125	70	130	30
n-Propylbenzene	70	130	65	140	30
2-Chlorotoluene	75	125	65	135	30
4-Chlorotoluene	75	130	65	135	30
1,3,5-Trimethylbenzene	75	130	65	140	30
tert-Butylbenzene	70	130	60	140	30
1,2,4-Trimethylbenzene	75	130	65	140	30
Sec-Butylbenzene	70	125	65	135	30
1,3-Dichlorobenzene	75	125	65	130	30
1,4-Dichlorobenzene	75	125	65	130	30
1,2-Dichlorobenzene	75	130	65	140	30
4-Isopropyltoluene	70	120	60	130	30
n-Butylbenzene	70	135	55	150	30
1,2,4-Trichlorobenzene	65	135	55	145	30
1,2,3-Trichlorobenzene	55	140	45	155	30
Hexachlorobutadiene	50	140	35	160	30
Naphthalene	55	140	40	150	30
1,2,3-Trichloropropane	75	125	65	130	30
1,2-Dibromo-3-chloropropane	50	130	35	145	30
Trichlorofluoromethane	60	145	45	160	30

1) For this VOC analyte list, up to three (3) sporadic MEs are allowed.

AML = Analytical Management Laboratories, Inc.
DoD = Department of Defense
LCS = Laboratory Control Limit
ME = Marginal Exceedence
MS/MSD = Matrix Spike/Matrix Spike Duplicate
QSM = Quality Systems Manual
REC = Percent Recovery
RPD = Relative Percent Difference

Table 12-5
QC Sample Acceptance Limits
SVOCs by SW-846 8270D (Soil/Sediment)
RI Addendum/FS
Former Schilling Air Force Base

Parameter	Control Limits LCS and MS/MSD		ME Control Limits LCS and MS/MSD		RPD Acceptable Limit
	Lower	Upper	Lower	Upper	Limit
	REC		REC		%
Bis(2-Chloroethyl)ether	40	105	25	115	30
Bis(2-chloroisopropyl)ether	20	115	10	130	30
2-Chlorophenol	45	105	35	115	30
1,3-Dichlorobenzene	40	100	30	110	30
1,4-Dichlorobenzene	35	105	25	115	30
1,2-Dichlorobenzene	45	95	35	105	30
Hexachloroethane	35	110	20	120	30
2-Methylphenol (o-Cresol)	40	105	30	115	30
3/4-Methylphenol (m/p-Cresol)	40	105	30	120	30
N-Nitroso-di-n-propylamine	40	115	30	125	30
Phenol	40	100	30	110	30
Bis(2-chloroethoxy)methane	45	110	30	120	30
4-Chloroaniline	10	95	0	110	30
4-Chloro-3-methylphenol	45	115	35	125	30
2,4-Dichlorophenol	45	110	35	120	30
2,4-Dimethylphenol	30	105	20	115	30
Hexachlorobutadiene	40	115	25	130	30
Isophorone	45	110	30	125	30
2-Methylnaphthalene	45	105	35	115	30
Naphthalene	40	105	30	120	30
Nitrobenzene	40	115	30	125	30
2-Nitrophenol	40	110	30	120	30
1,2,4-Trichlorobenzene	45	110	30	120	30
Acenaphthene	45	110	35	120	30
Acenaphthylene	45	105	35	115	30
2-Chloronaphthalene	45	105	35	115	30
4-Chlorophenyl-phenylether	45	110	35	120	30
Dibenzofuran	50	105	40	110	30
Diethylphthalate	50	115	40	125	30
Dimethylphthalate	50	110	40	120	30
2,4-Dinitrophenol	15	130	10	150	30
2,6-Dinitrotoluene	50	110	35	125	30
2,4-Dinitrotoluene	50	115	35	130	30
Fluorene	50	110	40	115	30
Hexachlorocyclopentadiene	40	115	25	130	30
2-Nitroaniline	45	120	30	130	30
3-Nitroaniline	25	110	15	125	30
4-Nitroaniline	35	115	20	125	30
4-Nitrophenol	15	140	10	160	30
2,4,6-Trichlorophenol	45	110	30	120	30
2,4,5-Trichlorophenol	50	110	40	120	30

Table 12-5
QC Sample Acceptance Limits
SVOCs by SW-846 8270D (Soil/Sediment)
RI Addendum/FS
Former Schilling Air Force Base

Parameter	Control Limits LCS and MS/MSD		ME Control Limits LCS and MS/MSD		RPD Acceptable Limit
	Lower	Upper	Lower	Upper	Limit
	REC		REC		%
Anthracene	55	105	45	115	30
Phenanthrene	50	110	40	120	30
4-Bromophenyl-phenylether	45	115	35	13	30
Di-n-butylphthalate	55	110	45	120	30
2-Methyl-4,6-dinitrophenol	30	135	10	155	30
Fluoranthene	55	115	45	125	30
Hexachlorobenzene	45	120	35	130	30
Pentachlorophenol	25	120	10	135	30
Benzo(a)anthracene	50	110	40	120	30
Chrysene	55	110	45	120	30
bis(2-ethylhexyl)phthalate	45	125	35	140	30
Butylbenzylphthalate	50	125	35	135	30
Pyrene	45	125	35	135	30
Di-n-octylphthalate	40	130	25	145	30
Indeno(1,2,3-cd)pyrene	40	120	25	135	30
Benzo(b)fluoranthene	45	115	35	125	30
Benzo(k)fluoranthene	45	125	30	135	30
Benzo(g,h,i)perylene	40	125	25	140	30
Benzo(a)pyrene	50	110	40	120	30
Dibenz(a,h)anthracene	40	125	25	140	30
Carbazole	45	115	30	130	30
Benzoic Acid	0	110	0	130	30
3,3'-Dichlorobenzidine	10	130	0	145	30
N-Nitrosodimethylamine	20	115	10	130	30
N-Nitrosodiphenylamine	50	115	40	125	30
Benzyl Alcohol	20	125	10	140	30

1) For this SVOC analyte list, up to three (3) sporadic MEs are allowed.

AML = Analytical Management Laboratories, Inc.
DoD = Department of Defense
LCS = Laboratory Control Limit
ME = Marginal Exceedence
MS/MSD = Matrix Spike/Matrix Spike Duplicate
QSM = Quality Systems Manual
REC = Percent Recovery
RPD = Relative Percent Difference

Table 12-6
QC Sample Acceptance Limits
SVOCs by SW-846 8270D (Water)
RI Addendum/FS
Former Schilling Air Force Base

Parameter	Control Limits LCS and MS/MSD		ME Control Limits LCS and MS/MSD		RPD Acceptable Limit
	Lower	Upper	Lower	Upper	Limit
	REC		REC		%
Bis(2-Chloroethyl)ether	35	110	25	120	30
Bis(2-chloroisopropyl)ether	25	130	10	150	30
2-Chlorophenol	35	105	25	115	30
1,3-Dichlorobenzene	30	100	20	110	30
1,4-Dichlorobenzene	30	100	20	110	30
1,2-Dichlorobenzene	35	100	20	115	30
Hexachloroethane	30	95	15	105	30
2-Methylphenol (o-Cresol)	40	110	25	120	30
3/4-Methylphenol (m/p-Cresol)	30	110	20	125	30
N-Nitroso-di-n-propylamine	35	130	20	145	30
Phenol	0	115	0	135	30
Bis(2-chloroethoxy)methane	45	105	35	115	30
4-Chloroaniline	15	110	10	125	30
4-Chloro-3-methylphenol	45	110	35	120	30
2,4-Dichlorophenol	50	105	40	115	30
2,4-Dimethylphenol	30	110	15	125	30
Hexachlorobutadiene	25	105	15	115	30
Isophorone	50	110	40	125	30
2-Methylnaphthalene	45	105	35	115	30
Naphthalene	40	100	30	115	30
Nitrobenzene	45	110	35	120	30
2-Nitrophenol	40	115	25	125	30
1,2,4-Trichlorobenzene	35	105	25	120	30
Acenaphthene	45	110	35	120	30
Acenaphthylene	50	105	40	115	30
2-Chloronaphthalene	50	105	40	115	30
4-Chlorophenyl-phenylether	50	110	40	120	30
Dibenzofuran	55	105	45	115	30
Diethylphthalate	40	120	30	130	30
Dimethylphthalate	25	125	10	145	30
2,4-Dinitrophenol	15	140	10	160	30
2,6-Dinitrotoluene	50	115	35	130	30
2,4-Dinitrotoluene	50	120	40	130	30
Fluorene	50	110	40	120	30
Hexachlorocyclopentadiene	25	105	15	115	30
2-Nitroaniline	50	115	35	125	30
3-Nitroaniline	20	125	10	145	30
4-Nitroaniline	35	120	20	130	30
4-Nitrophenol	0	125	0	145	30
2,4,6-Trichlorophenol	50	115	40	125	30
2,4,5-Trichlorophenol	50	110	40	120	30

Table 12-6
QC Sample Acceptance Limits
SVOCs by SW-846 8270D (Water)
RI Addendum/FS
Former Schilling Air Force Base

Parameter	Control Limits LCS and MS/MSD		ME Control Limits LCS and MS/MSD		RPD Acceptable Limit
	Lower	Upper	Lower	Upper	Limit
	REC		REC		%
Anthracene	55	110	45	120	30
Phenanthrene	50	115	40	130	30
4-Bromophenyl-phenylether	50	115	40	125	30
Di-n-butylphthalate	40	130	25	145	30
2-Methyl-4,6-dinitrophenol	55	115	45	125	30
Fluoranthene	55	115	45	125	30
Hexachlorobenzene	50	110	40	120	30
Pentachlorophenol	40	115	25	130	30
Benzo(a)anthracene	55	110	45	120	30
Chrysene	55	110	45	120	30
bis(2-ethylhexyl)phthalate	40	125	30	140	30
Butylbenzylphthalate	45	115	35	130	30
Pyrene	50	130	35	140	30
Di-n-octylphthalate	35	135	20	155	30
Indeno(1,2,3-cd)pyrene	45	125	30	140	30
Benzo(b)fluoranthene	45	120	35	130	30
Benzo(k)fluoranthene	45	125	30	135	30
Benzo(g,h,i)perylene	40	125	25	135	30
Benzo(a)pyrene	55	110	45	120	30
Dibenz(a,h)anthracene	40	125	30	140	30
Carbazole	50	115	35	130	30
Benzoic Acid	0	125	0	150	30
3,3'-Dichlorobenzidine	20	110	10	125	30
N-Nitrosodimethylamine	25	110	10	125	30
N-Nitrosodiphenylamine	50	110	35	120	30
Benzyl Alcohol	30	110	15	125	30

1) For this SVOC analyte list, up to three (3) sporadic MEs are allowed.

AML = Analytical Management Laboratories, Inc.
DoD = Department of Defense
LCS = Laboratory Control Limit
ME = Marginal Exceedence
MS/MSD = Matrix Spike/Matrix Spike Duplicate
QSM = Quality Systems Manual
REC = Percent Recovery
RPD = Relative Percent Difference

Table 12-7
QC Sample Acceptance Limits
MEE by RSK-175 (Water)
RI Addendum/FS
Former Schilling Air Force Base

Parameter	Control Limits LCS		Control Limits MS/MSD		RPD Acceptable Limit
	Lower	Upper	Lower	Upper	Limit
	REC		REC		%
Methane	70	130	70	130	30
Ethane	70	130	70	130	30
Ethene	70	130	70	130	30

1) DoD QSM control limits are not available for this analysis.

AML = Analytical Management Laboratories, Inc.
DoD = Department of Defense
LCS = Laboratory Control Limit
ME = Marginal Exceedence
MS/MSD = Matrix Spike/Matrix Spike Duplicate
QSM = Quality System Manual
REC = Percent Recovery
RPD = Relative Percent Difference

Table 12-8
QC Sample Acceptance Limits
Metals by SW-846 6020A & 7471A (Soil/Sediment)
RI Addendum/FS
Former Schilling Air Force Base

Parameter	Control Limits LCS		Control Limits MS/MSD		RPD Acceptable Limit
	Lower	Upper	Lower	Upper	Limit
	REC		REC		%
Arsenic	80	120	75	125	20
Barium	80	120	75	125	20
Cadmium	80	120	75	125	20
Chromium	80	120	75	125	20
Lead	80	120	75	125	20
Mercury	80	120	80	120	20
Selenium	80	120	75	125	20
Silver	80	120	75	125	20

1) AML's control limits for LCS are the same as DoD QSM control limits.

AML = Analytical Management Laboratories, Inc.
DoD = Department of Defense
LCS = Laboratory Control Limit
ME = Marginal Exceedence
MS/MSD = Matrix Spike/Matrix Spike Duplicate
QSM = Quality Systems Manual
REC = Percent Recovery
RPD = Relative Percent Difference

Table 12-9
QC Sample Acceptance Limits
Metals by SW-846 6020A & 7470A (Water)
RI Addendum/FS
Former Schilling Air Force Base

Parameter	Control Limits LCS		Control Limits MS/MSD		RPD Acceptable Limit
	Lower	Upper	Lower	Upper	Limit
	REC		REC		%
Arsenic	80	120	75	125	20
Barium	80	120	75	125	20
Cadmium	80	120	75	125	20
Chromium	80	120	75	125	20
Iron	80	120	75	125	20
Lead	80	120	75	125	20
Mercury	80	120	80	120	20
Selenium	80	120	75	125	20
Silver	80	120	75	125	20

1) AML's control limits for LCS are the same as DoD QSM control limits.

AML = Analytical Management Laboratories, Inc.
DoD = Department of Defense
LCS = Laboratory Control Limit
ME = Marginal Exceedence
MS/MSD = Matrix Spike/Matrix Spike Duplicate
QSM = Quality Systems Manual
REC = Percent Recovery
RPD = Relative Percent Difference

Table 12-10
QC Sample Acceptance Limits
Natural Attenuation Indicator Parameters (Water)
RI Addendum/FS
Former Schilling Air Force Base

Parameter	Control Limits LCS		Control Limits MS/MSD		RPD Acceptable Limit	Lab Duplicate RPD Acceptable Limit
	REC		REC		Limit	Limit
	Lower	Upper	Lower	Upper	%	%
Metals (SW-846 6010B)						
Iron	See Table 12-9 (Metals by SW-846 6020A and 7470A [Water])					
Anions (EPA Method 300.0)						
Chloride	85	115	80	120	20	NA
Nitrite	85	115	80	120	20	NA
Nitrate	85	115	80	120	20	NA
Sulfate	85	115	80	120	20	NA
Sulfide (EPA Method 376.1)						
Sulfide	70	130	70	130	30	25
TOC (SW-846 9060)						
Total Organic Carbon	70	130	70	130	30	25

1) DoD QSM control limits are not available for these analyses.
2) Control limits are obtained from AML's SOPs.

AML = Analytical Management Laboratories, Inc.
DoD = Department of Defense
LCS = Laboratory Control Limit
ME = Marginal Exceedence
MS/MSD = Matrix Spike/Matrix Spike Duplicate
NA = Not Applicable
QSM = Quality System Manual
REC = Percent Recovery
RPD = Relative Percent Difference

Table 12-11
QC Sample Acceptance Limits
VOC Surrogate Spikes (Water/Soil)
RI Addendum/FS
Former Schilling Air Force Base

Surrogate Compound	QC Limits for REC			
	Water		Soil	
	Lower	Uppper	Lower	Uppper
4-Bromofluorobenzene	75	120	85	120
Dibromofluoromethane	85	115	85	119
Toluene-d8	85	120	85	115
1,2-Dichloroethane-d4	70	120	78	129

1) MEs are not applicable for surrogate spikes.

ME = Marginal Exceedences
QC = Quality Control
REC = Percent Recovery

Table 12-12
QC Sample Acceptance Limits
SVOC Surrogate Spikes (Water/Soil)
RI Addendum/FS
Former Schilling Air Force Base

Surrogate Compound	QC Limits for REC			
	Water		Soil	
	Lower	Upper	Lower	Upper
2-Flourophenol	20	110	35	105
Phenol-d5	10	115	40	100
Nitrobenzene-d5	40	110	35	100
2-Fluorobiphenyl	50	110	45	105
2,4,6-Tribromophenol	40	125	35	125
Terphenyl-d14	50	135	30	125

1) MEs are not applicable for surrogate spikes.

ME = Marginal Exceedences
QC = Quality Control
REC = Percent Recovery

Table 12-13
Cross Reference for Laboratory Reporting Limits
RI Addendum/FS
Former Schilling Air Force Base

AML	UFP QAPP Manual	DoD QSM	Definition
PQL	QL	LOQ	The minimum concentration of a constituent that can be routinely quantified by the lab. This is commonly known as the reporting limit.
LLR	MDL	LOD	A statistically-derived value that represents the 99 percent confidence level that the reported signal is different from a blank sample. These are lower than the QL/LOQ/PQL.

AML = Analytical Management Laboratories, Inc.
DoD QSM = Department of Defense Quality Systems Manual for Environmental Laboratories
LLR = Lowest Level of Reporting
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit
QL = Quantitation Limit
PQL = Practical Quantitation Limit
UFP QAPP = Uniform Federal Policy for Quality Assurance Project Plans

Table 24-1
Summary of Calibration and Quality Control Procedures for VOCs by SW-846 8260B
RI Addendum/FS
Former Schilling Air Force Base

METHOD	APPLICABLE PARAMETER	QC CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTIONS
SW-846 8260B	VOCs	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	Average RF for SPCCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. % RSD for RFs for VOCs ≤ 30% and one option below: *option 1 linear-* RSD for each analytes ≤15% *option 2 linear-* least squares regression r > 0.995 for analytes not meeting RSD criteria *option 3 non-linear-* COD r^2 ≥ 0.990 (6 points shall be used for second order, 7 points shall be used for third order) for analytes not meeting RSD criteria	Correct problem then repeat initial calibration if an SPCC or CCC does not meet criteria. Also repeat if more than 5 other compounds require application of R according to flagging criteria.
		Second-source calibration verification	Once per five-point initial calibration	All analytes within ± 25% of expected value	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.
		Retention time window verified for each analyte and surrogate	Each sample	Position shall be set using the midpoint standard of the initial calibration curve. RRT of the analyte within± 0.06 RRT units of the RRT	Correct problem then reanalyze all samples analyzed since the last retention time check
		Calibration verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF≥0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. CCCs ≤ 20% difference (when using RFs) or drift (when using least squares regression or nonlinear calibration)	Correct problem then repeat initial calibration if an SPCC or CCC does not meet the criteria. Also repeat if more than 5 compounds exceed 30%.

Table 24-1
Summary of Calibration and Quality Control Procedures for VOCs by SW-846 8260B
RI Addendum/FS
Former Schilling Air Force Base

METHOD	APPLICABLE PARAMETER	QC CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTIONS
SW-846 8260B	VOCs	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria based upon each laboratory and/or DoD limits. Limits updated annually.	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria. Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.
		Internal Standards	Immediately after or during data acquisition for each sample	Retention time ± 30 seconds from retention time of the midpoint standard in the initial calibration.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
				EICP area within -50% to +100% of CCV	
		Confirmation of positive detections	Each positive detection	Confirmation of positive detection(s)	If positive detection not confirmed in reanalysis, analyze standard(s), correct problem, and reanalyze samples.
		Method blank	One per analytical batch (20 samples or less)	No analytes detected ≥ 1/2X PQL, except methylene chloride and acetone-must be less than or equal to the PQL.	Correct problem then reanalyze. If still out then reprep and analyze method blank and all samples processed with the contaminated blank.
		LCS for all analytes	One LCS per analytical batch; may be analyzed in duplicate	Tables 12-3 (Soil/Sediment) and 12-4 (Water). QC acceptance criteria for control and marginal exceedence based upon DoD Criteria and/or laboratory's limits. [a] Limits updated annually.	Correct problem then reanalyze the LCS and all samples in the associated prepatory batch for failed analytes.

Table 24-1
Summary of Calibration and Quality Control Procedures for VOCs by SW-846 8260B
RI Addendum/FS
Former Schilling Air Force Base

METHOD	APPLICABLE PARAMETER	QC CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTIONS
SW-846 8260B	VOCs	MS/MSD	One MS/MSD per 20 samples per matrix	Tables 12-3 (Soil/Sediment) and 12-4 (Water). QC acceptance criteria for control and marginal exceedence based upon DoD Criteria and/or laboratory's limits. [a] Limits updated annually.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.
		Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description	Retune instrument and verify Proper tuning must be verified prior to sample analysis
		Surrogate spike	Every sample, spiked sample, standard, and method blank	Table 12-11. QC acceptance criteria based upon DoD Criteria and/or laboratory's limits. Limits updated annually.	Correct problem then re-extract and analyze sample(s) for failed surrogate(s).
		MDL study	Performed when significant instrument change occurs. Verified quarterly per DoD QSM.	See 40 CFR 136B. MSL verification checks must produce a signal at least 3 times the instrument's noise level	None
		Results reported between MDL and PQL	None	Report as estimated ("J" flag)	None

a = The limits and MDLs (LOD) listed in this QAPP were the most current at the time this document was written. These limits and MDLs (LOD) are subject to change depending upon the start time of this project. The most current MDLs (LOD) and limits will be used for this project and may or may not be those listed in the enclosed tables. Control limits and marginal exceedence limits for LCS recoveries used by the laboratory are based on DoD QSM, final version 3, Jan. 2006.

BFB = Bromofluorobenzene
CA = Corrective Action
CCC = Calibration Check Compound
CCV = Continuing Calibration Verification
CFR = Code Federal Regulations
COD = Coefficient of Determination
DoD = Department of Defense
EICP = Extracted Ion Current Profile
GC = Gas Chromatography
LCS = Laboratory Control Sample
LOD = Limit of Detection
MDL = Method Detection Limit (Equivalent to LOD)
MS/MSD = Matrix Spike/Matrix Spike Duplicate
QC = Quality Control
QSM = *Quality System Manual for Environmental Laboratories, Version 3, Final*
PQL = Practical Quantitation Limit (Equivalent to LOQ)
RF = Response Factor
RRT = Relative Retention Time
RSD = Relative Standard Deviation
SPCC = System Performance Check Compound
VOC = Volatile Organic Compound

Table 24-2
Summary of Calibration and Quality Control Procedures for SVOCs by SW-846 8270D
RI Addendum/FS
Former Schilling Air Force Base

METHOD	APPLICABLE PARAMETER	QC CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTIONS
SW-846 8270D	SVOCs	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	Average RF for SPCCs ≥ 0.050 and %RSD for RFs for CCCs ≤ 30% and one option below: *option 1 linear-* Response factor method for analytes with mean RSD ≤15% *option 2 linear-* least squares regression r > 0.995 for analytes not meeting RSD criteria *option 3 non-linear-* COD r^2 ≥ 0.990 (6 points shall be used for second order, 7 points shall be used for third order) for analytes not meeting RSD criteria	Correct problem then repeat initial calibration if an SPCC or CCC does not meet criteria. Also repeat if more than 5 other compounds require application of R according to flagging criteria.
		Second-source calibration verification	Once per five-point initial calibration	All analytes within ± 25% of expected value	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.
		Retention time window verified for each analyte and surrogate	Each sample	RRT of the analyte within± 0.06 RRT units of the RRT	Correct problem then reanalyze all samples analyzed since the last retention time check
		Calibration verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF≥0.050; and CCCs ≤ 20% difference (when using RFs) or drift (when using least squares regression or non-linear calibration)	Correct problem then repeat initial calibration if an SPCC or CCC does not meet the criteria. Also repeat if more than 5 compounds exceed 30%.

Table 24-2
Summary of Calibration and Quality Control Procedures for SVOCs by SW-846 8270D
RI Addendum/FS
Former Schilling Air Force Base

METHOD	APPLICABLE PARAMETER	QC CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTIONS
SW-846 8270C	SVOCs	Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria based upon each laboratory and/or DoD limits. Limits updated annually.	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria
		Internal Standards	Immediately after or during data acquisition for each sample	Retention time ± 30 seconds from retention time in the CCV	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
				EICP area within -50% to +100% of CCV	
		Method blank	One per analytical batch (20 samples or less)	No analytes detected ≥ 1/2X PQL, except analytes ending in "phthalate" must be less than or equal to the PQL.	Correct problem then reanalyze. If still out then reprep and analyze method blank and all samples processed with the contaminated blank
		LCS for all analytes	One LCS per analytical batch; may be analyzed in duplicate	Table 12-5 (Soil/Sediment) and 12-6 (Water). QC acceptance criteria for control and marginal exceedence based upon DoD Criteria and/or laboratory's limits. [a] Limits updated annually.	Correct problem then reanalyze the LCS and all samples in the associated prepatory batch for failed analyte.
		MS/MSD	One MS/MSD per 20 samples per matrix	Table 12-5 (Soil/Sediment) and 12-6 (Water). QC acceptance criteria based upon DoD Criteria and/or laboratory limits. Limits updated annually	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.
		Check of mass spectral ion intensities using BFB (Tuning)	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description	Retune instrument and verify Proper tuning must be verified prior to sample analysis

Table 24-2
Summary of Calibration and Quality Control Procedures for SVOCs by SW-846 8270D
RI Addendum/FS
Former Schilling Air Force Base

METHOD	APPLICABLE PARAMETER	QC CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTIONS
SW-846 8270C	SVOCs	Surrogate spike	Every sample, spiked sample, standard, and method blank	Table 12-11. QC acceptance criteria based upon DoD Criteria and/or laboratory's limits. Limits updated annually.	Correct problem then re-extract and analyze sample(s) for failed surrogates.
		MDL study	Performed when significant instrument change occurs. Verified quarterly per DoD QSM.	See 40 CFR 136B. MSL verification checks must produce a signal at least 3 times the instrument's noise level	None
		Results reported between MDL and PQL	None	Report as estimated ("J" flag)	None

a = The limits and MDLs (LOD) listed in this QAPP were the most current at the time this document was written. These limits and MDLs (LOD) are subject to change depending upon the start time of this project. The most current MDLs (LOD) and limits will be used for this project and may or may not be those listed in the enclosed tables. Control limits and marginal exceedence limits for LCS recoveries used by the laboratory are based on DoD QSM, final version 3, Jan. 2006.

BFB = Bromofluorobenzene
CA = Corrective Action
CCC = Calibration Check Compound
CCV = Continuing Calibration Verification
CFR = Code Federal Regulations
COD = Coefficient of Determination
DoD = Department of Defense
EICP = Extracted Ion Current Profile
GC = Gas Chromatography
LCS = Laboratory Control Sample
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit (Equivalent to LOD)
MS/MSD = Matrix Spike/Matrix Spike Duplicate
QC = Quality Control
QSM = *Quality System Manual for Environmental Laboratories, Version 3, Final*
PQL = Practical Quantitation Limit (Equivalent to LOQ)
RF = Response Factor
RRT = Relative Retention Time
RSD = Relative Standard Deviation
SPCC = System Performance Check Compound
SVOC = Semivolatile Organic Compound

Table 24-3
Summary of Calibration and Quality Control Procedures for MEE by RSK-175
RI Addendum/FS
Former Schilling Air Force Base

METHOD	APPLICABLE PARAMETER	QC CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTIONS
RSK-175	MEE	Five-point initial calibration (low point at the reporting limit)	Initial calibration prior to sample analysis	RF method for analytes with %RSD <20%; and/or ≥ 0.990 for linear regression	Correct problem then repeat initial calibration
		Second-source calibration verification	Once per three point initial calibration	All analytes within ± 20% of expected value	Correct problem then repeat initial calibration
		Continuing calibration verification	Daily, before sample analysis, and after every 20 samples thereafter, and after the last sample in the sequence	All analytes within ± 20% of expected value	Reanalyze to verify. If still deficient, correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample	Once per analyst	All analytes within ± 20% of expected value	Recalculate results; locate and fix problem with system, rerun demonstration for those analytes that did not meet criteria
		Method Blank	Once per analytical batch (20 samples or less)	No analytes detected >1/2 of PQL, with the exception of methane.	1. Correct problem then reanalyze. If still out then investigate source of contamination 2. Reprep and analyze method blank and all samples processed with the contaminated blank
		LCS for all analytes	One LCS per analytical batch; LCS may be analyzed in duplicate	Table 12-7. QC acceptance criteria based upon each laboratory's limits[a]. Limits updated annually.	Correct problem then reanalyze. If still out then reprep and analyze all affected QC and field samples
		MS/MSD	One MS/MSD per 20 samples per matrix	Table 12-7. QC acceptance criteria based upon each laboratory's limits[a]. Limits updated annually.	None
		Surrogate spike	Not applicable	Not applicable	None
		MDL Study	Performed when significant instrument change occurs. Verified quarterly per DoD QSM.	None	None
		Results reported between MDL and PQL	None	Report as estimated value ("J" flag)	None

Table 24-3
Summary of Calibration and Quality Control Procedures for MEE by RSK-175
RI/FS Addendum
Former Schilling Air Force Base

a = The limits and MDLs (LOD) listed in this QAPP were the most current at the time this document was written. These limits and MDLs (LOD) are subject to change depending upon the start time of this project. The most current MDLs (LOD) and limits will be used for this project and may or may not be those listed in the enclosed tables. Control limits and marginal exceedence limits for LCS recoveries used by the laboratory are based on DoD QSM, final version 3, Jan. 2006.

DoD = Department of Defense
MDL = Method Detection Limit (Equivalent to LOD)
LCS = Laboratory Control Sample
LOD = Limit of Detection
LOQ = Limit of Quantitation
MEE = Methane, Ethane, Ethene
MS/MSD = Matrix Spike/Matrix Spike Duplicate
QC = Quality Control
QSM = *Quality System Manual for Environmental Laboratories, Version 3, Final*
PQL = Practical Quantitation Limit (Equivalent to LOQ)
RF = Response Factor
RSD = Relative Standard Deviation

Table 24-4
Summary of Calibration and Quality Control Procedures for Metals by SW-846 6020A
RI Addendum/FS
Former Schilling Air Force Base

METHOD	APPLICABLE PARAMETER	QC CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTIONS
SW-846 6020A	ICP-MS Metals	Initial calibration minimum 1 high standard and a blank (Standardization) or 3 standards and a blank)	Daily initial calibration prior to sample analysis	N/A for one point. Correlation coefficient ≥ 0.995 for linear regression	Correct problem then repeat initial calibration
		Verification of Standardization	Daily prior to analyzing High Level Standard	Mass calibration ≤ 0.1 amu from the true value; Resolution < 0.9 amu full width at 10% peak height; For stability, RSD ≤ 5% for at least four replicate samples	Correct problem and repeat standardization
		Verification of the dynamic linear range (HLSTD)	Daily after Standardization	All analytes within ±10% of expected value	Correct problem then repeat HLSTD or analyze samples exceeding the highest calibration standard under appropriate dilution.
		ICV (second source ICV)	Daily after initial calibration, prior to beginning a sample run	All analytes within ±10% of expected value	Correct problem and verify second source standard. Rerun second source verification. Of that fails, correct problem and then repeat initial calibration.
		ICB	Daily after ICV	No analytes detected > ½ PQL	Correct potential carryover problem and reanalyze ICV and ICB.
		MQL Check or CRI (Contract required detection limit check for ICP in Superfund)	Daily after ICB and before ICSA and ICSAB sample analyses	All analytes within ±20% of expected value	Correct problem and reanalyze samples. Adjust PQL for samples, if acceptable.
		ICS (ICSA and ICSAB); additional ICS samples, as necessary	At the beginning and end of an analytical run	Absolute value of concentration for all non-spiked analytes <2x MDL Within ± 20% of expected value for ICSAB	Correct problem; adjust IEC and reanalyze ICS samples; reanalyze affected samples.
		CCV and ECV	Before sample analysis, after every 10 samples, and at the end of sample analysis	All analytes within ±10% of expected value	Correct problem and reanalyze samples before and after failed CV after a passing CV.
		CCBs, and FCB	After every calibration verification. Before beginning a sample run, after every 10 samples and at end of the analysis sequence	No analytes detected >2x PQL	Correct problem and reanalyze samples before and after failed CB after a passing CB.
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria based upon DoD Criteria and/or laboratory's limits. Limits updated annually.	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria

Table 24-4
Summary of Calibration and Quality Control Procedures for Metals by SW-846 6020A
RI Addendum/FS
Former Schilling Air Force Base

METHOD	APPLICABLE PARAMETER	QC CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTIONS
		Method blank	One per analytical batch, (20 samples or less)	No analytes detected ≥ ½ PQL	Correct problem then reanalyze, If still out then reprep and analyze method blank and all samples processed with the contaminated blank
SW-846 6020A	ICP-MS Metals	LCS for the analyte	One LCS per analytical batch (20 samples)	Tables 12-8 (Soil/Sediment) and 12-9 (Water). QC acceptance criteria based upon DoD Criteria and/or laboratory's limits[a]. Limits updated annually.	Correct problem then reanalyze. If still out then reprep and analyze the LCS and all samples in the affected analytical batch for failed analytes
		Dilution test (SD)	Each prep batch	1:5 dilution must agree within ± 10% of the original determination if sample result is greater than 50X MDL.	Perform PDS test
		PDS test	Each prep batch	Recoveries within 75 to 125% (80% to 120% criteria will be used per recent SW Method Revision)	Check another sample. Perform MSA or reanalyze under dilution when MS, MSD, SD and PDS fail for the analyte.
		MSA	When matrix interference is suspected	N/A	N/A
		MS/MSD	One MS/MSD per 20 samples per matrix	Tables 12-8 (Soil/Sediment) and 12-9 (Water). QC acceptance criteria based upon DoD Criteria and/or laboratory limits[a]. Limits updated annually.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken
		MDL study	Performed when significant instrument change occurs. Verified quarterly per DoD QSM.	See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument noise level	Run MDL verification check at higher level and set MDL higher or reconduct MDL study
		IDL study	At initial set-up and after significant change	Detection limits established shall be ≤ MDL	N/A
		IS	Every sample	IS intensity within 30-120% of intensity of the IS in the initial calibration	Perform corrective action as described in SW-846 6020A
		Results reported between MDL and PQL	None	Report as estimated ("J" flag)	None

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CB = Calibration blank
CCB = Continuing Calibration Blank
CCV = Continuing Calibration Verification
CV = Calibration Verification
DoD = Department of Defense
FCB = Ending Calibration Blank
FCV = Ending Calibration Verification
ICB = Initial Calibration Blank
ICP = Inductively Coupled Plasma
ICS = Interference Check Solution
LCS = Laboratory Control Sample
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit (Equivalent to LOD)
MQL = Method Quantitation Limit
MS/MSD = Matrix Spike/Matrix Spike Duplicate
MSA = Method of Standard Addition
N/A = Not Applicable
QC = Quality Control
QSM = *Quality System Manual for Environmental Laboratories, Version 3, Final*

Table 24-5
Summary of Calibration and Quality Control Procedures for Mercury by SW-846 7470A/7471A
RI Addendum/FS
Former Schilling Air Force Base

METHOD	APPLICABLE PARAMETER	QC CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTIONS
SW-846 7470A (Water) SW-846 7471A (Soil)	Mercury	Initial calibration minimum 5 standards and a calibration blank	Daily initial calibration prior to sample analysis	Correlation coefficient ≥ 0.995 for linear regression	Correct problem then repeat initial calibration
		Verification of Standardization	Daily prior to analyzing High Level Standard	± 10% of true value	Correct problem and repeat standardization
		ICV (second source ICV)	Daily after initial calibration and prior to sample analysis	All analytes within ±10% of expected value	Correct problem then repeat initial calibration.
		ICB	Daily after ICV	No analytes detected > ½ PQL	Correct potential carryover problem and reanalyze ICV and ICB.
		CCV and ECV	Before sample analysis, after every 10 samples, and at the end of sample analysis	All analytes within ±20% of expected value	Correct problem and reanalyze samples before and after failed CV after a passing CV.
		CCBs and FCB	After every calibration verification	No analytes detected > 2xMDL	Correct problem and reanalyze previous 10 samples before and after failed CB after a passing CB.
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria based upon DoD Criteria and/or laboratory's limits. Limits updated annually.	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria
		Method blank	One per analytical batch, (20 samples or less)	No analytes detected ≥ ½ PQL	Correct problem then reanalyze, If still out then reprep and analyze method blank and all samples processed with the contaminated blank
		LCS for the analyte	One LCS per analytical batch (20 samples)	Tables 12-8 (Soil/Sediment) and 12-9 (Water). QC acceptance criteria based upon DoD Criteria and/or laboratory's limits[a]. Limits updated annually.	Correct problem then reanalyze. If still out then reprep and analyze the LCS and all samples in the affected analytical batch for the failed analytes
		Dilution test (SD)	Each prep batch	1:5 dilution must agree within ± 10% of the original determination if sample result is greater than 25X MDL.	Perform matrix spike
		MS/MSD	One MS/MSD per 20 samples per matrix	Tables 12-8 (Soil/Sediment) and 12-9 (Water). QC acceptance criteria, based upon DoD Criteria and/or laboratory limits[a]. Limits updated annually.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken

Table 24-5
Summary of Calibration and Quality Control Procedures for Mercury by SW-846 7470A/7471A
RI Addendum/FS
Former Schilling Air Force Base

METHOD	APPLICABLE PARAMETER	QC CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTIONS
SW-846 7470A (Water) SW-846 7471A (Soil)	Mercury	MDL study	Performed when significant instrument change occurs. Verified quarterly per DoD QSM.	See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument noise level	Run MDL verification check at higher level and set MDL higher or reconduct MDL study
		MSA	When matrix interference is suspected	N/A	N/A
		Results reported between MDL and PQL	None	Report as estimated ("J" flag)	None

a = The limits and MDLs (LOD) listed in this QAPP were the most current at the time this document was written. These limits and MDLs (LOD) are subject to change depending upon the start time of this project. The most current MDLs (LOD) and limits will be used for this project and may or may not be those listed in the enclosed tables. Control limits and marginal exceedence limits for LCS recoveries used by the laboratory are based on DoD QSM, final version 3, Jan. 2006.

CB = Calibration blank
CCB = Continuing Calibration Blank
CCV = Continuing Calibration Verification
CV = Calibration Verification
DoD = Department of Defense
FCB = Ending Calibration Blank
FCV = Ending Calibration Verification
ICB = Initial Calibration Blank
ICP = Inductively Coupled Plasma
ICS = Interference Check Solution
ICV = Initial Calibration Verification
LCS = Laboratory Control Sample
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit (Equivalent to LOD)
MQL = Method Quantitation Limit
MS/MSD = Matrix Spike/Matrix Spike Duplicate
N/A = Not Applicable
QC = Quality Control
QSM = *Quality System Manual for Environmental Laboratories, Version 3, Final*
PDS = Post Digestion Spike
PQL = Practical Quantitation Limit (Equivalent to LOQ)
SD = Dilution Test

Table 24-6
Summary of Calibration and Quality Control Procedures for Anions by EPA Method 300.0
RI Addendum/FS
Former Schilling Air Force Base

METHOD	APPLICABLE PARAMETER	QC CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
EPA Method 300.0	Cl^-, NO_2^-, NO_3^-, SO_4^{-2}	Multipoint calibration for all analytes (minimum 3 standards and one calibration blank)	Initial calibration prior to sample analysis	Correlation coefficient 0.995 for linear regression	Correct problem then repeat initial calibration
		Second-source calibration verification	Once per multipoint calibration	All analytes within ±10% of expected value	Correct problem then repeat initial calibration
		Retention time window verified for each analyte	Each initial calibration and Calibration verifications	± 3x standard deviation for each analyte retention time over 24 hour period	Correct problem then reanalyze all samples analyzed since the last retention time check
		Retention time window position establishment for each analyte	Once per multipoint calibration	Position shall be at midpoint of initial calibration curve	N/A
		Retention time window verification for each analyte	Each calibration standard	Analyte within established window	Correct problem, then reanalyze all samples analyzed since the last retention time check. If they fail, redo initial calibration and reset retention time window
		Initial calibration verification	Daily, before sample analysis or when elutent is changed and with every batch of samples	All analytes within ±10% of expected value and retention times within appropriate windows	Correct problem then repeat initial calibration
		Calibration verification	After every 10 samples and at the end of the analysis sequence	Instrument response within ± 10% of expected response	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria based upon each laboratory's limits[a]. Limits updated annually.	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria
		Method blank	One per analytical batch	No analytes detected > ½ PQL	Correct problem then reanalyze. If still out then reprep and analyze method blank and all samples processed with the contaminated blank

Table 24-6
Summary of Calibration and Quality Control Procedures for Anions by USEPA 300.0
RI Addendum/FS
Former Schilling Air Force Base

METHOD	APPLICABLE PARAMETER	QC CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
		LCS for all analytes	One LCS per analytical batch	Table 12-10. QC acceptance criteria based upon laboratory's limits[a]. Limits updated annually.	Correct problem then reanalyze. if still out then reprep and analyze the LCS and all samples in the affected analytical batch for failed analyte
		MS/MSD	One MS/MSD per 20 samples per matrix	Table 12-10. QC acceptance criteria based upon DoD Criteria and/or laboratory's limits[a]. Limits updated annually.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken
		MDL study	Performed when significant instrument change occurs. Verified quarterly per DoD QSM.	See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument noise level	Run MDL verification check at higher level and set MDL higher or reconduct MDL study
		Sample Duplicate (Replicate)	One per every 10 samples	%D ≤ 10% (between sample and sample duplicate)	Correct problem and reanalyze sample and duplicate
		Results reported between MDL and PQL	None	Code as estimated ("J" flag) value	None

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DoD = Department of Defense
LCS = Laboratory Control Sample
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit (Equivalent to LOD)
MQL = Method Quantitation Limit
MS/MSD = Matrix Spike/Matrix Spike Duplicate
PQL = Practical Quantitation Limit (Equivalent to LOQ)
QC = Quality Control
QSM = *Quality System Manual for Environmental Laboratories, Version 3, Final*

Table 24-7
Summary of Calibration and Quality Control Procedures for Sulfide by EPA 376.1
RI Addendum/FS
Former Schilling Air Force Base

METHOD	APPLICABLE PARAMETER	QC CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
EPA Method 376.1	Sulfide	Multipoint calibration using undistilled standards for color reaction or Titrimetry	Initial calibration prior to sample analysis	Correlation Coefficient ≥ 0.995 for linear regression for color reaction	Correct problem then repeat initial calibration
		ICV with undistilled standard, second source	Once per multipoint calibration	Observed result within ±15% of expected value	Correct problem then repeat initial calibration
		CCVs with undistilled standard (same source)	Initially after ICV, after every 10 samples, and at the end of the analysis sequence	Observed result within ±10% of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification.
		ICBs and CCBs undistilled	Immediately after ICV and CCV standards	Less than ½ of PQL	Correct problem and reanalyze
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	QC acceptance criteria based upon each laboratory's limits[a]. Limits updated annually.	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria
		Method blank	One per analytical batch	Less than ½ of PQL	Correct problem then reanalyze. If still out then reprep and analyze method blank and all samples processed with the contaminated blank
		LCS	One LCS samples per analytical batch	Table 12-10	Correct problem then reanalyze. if still out then reprep and analyze the LCS and all samples in the affected analytical batch
		MS/MSD	One MS/MSD per 20 samples per matrix	Table 12-10	None
		MDL study	Performed when significant instrument change occurs. Verified quarterly per DoD QSM.	None	None
		Results reported between MDL and PQL	None	Report as estimated value ("J" flag)	None

Table 24-7
Summary of Calibration and Quality Control Procedures for Sulfide by EPA Method 376.1
RI Addendum/FS
Former Schilling Air Force Base

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CCB = Continuing Calibration Blank
CCV = Continuing Calibration Verification
DoD = Department of Defense
ICB = Initial Calibration Blank
ICV = Initial Calibration Verification
LCS = Laboratory Control Sample
LLR = Lowest Level for Reporting
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit (Equivalent to LOD)
MS/MSD = Matrix Spike/Matrix Spike Duplicate
PQL = Practical Quantitation Limit (Equivalent to LOQ)
QC = Quality Control
QSM = *Quality System Manual for Environmental Laboratories, Version 3, Final*

Table 24-8
Summary of Calibration and Quality Control Procedures for TOC by SW-846 9060
RI Addendum/FS
Former Schilling Air Force Base

METHOD	APPLICABLE PARAMETER	QC CHECK	MINIMUM FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
SW-846 9060	Total Organic Carbon	Multipoint calibration (5 levels and one blank) ion	Initial calibration prior to sample analysis	Correlation coefficient ≥ 0.995 for linear regression	Correct problem then repeat initial calibration
		ICV with second source standard	Once per multipoint calibration	Observed result within ±15% of expected value	Correct problem then repeat initial calibration
		CCVs with same source standard	Initially after ICV, after every 10 samples, and at the end of the nalysis sequence	Observed result within ±10% of expected value	Correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification
		ICBs and CCBs	Immediately after ICV and CCV standards	Less than ½ of PQL	Correct problem and reanalyze
		Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample	Once per analyst	Observed result within ±30% of expected value.	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria.
		Method blank	One per analytical batch	No analytes detected > ½ PQL	Correct problem then reanalyze. If still out then reprep and analyze method blank and all samples processed with the contaminated blank
		LCS (same source)	One LCS sample per analytical batch	Table 12-10. QC acceptance criteria based upon each laboratory's limits[a]. Limits updated annually.	Correct problem then reanalyze. If still out then reprep and analyze the LCS and all samples in the affected analytical batch.
		MS/MSD	One MS/MSD per 20 samples per matrix	Table 12-10. QC acceptance criteria based upon each laboratory's limits[a]. Limits updated annually.	None
		MDL study	Performed when significant instrument change occurs. Verified quarterly per DoD QSM.	None	None
		Results reported between MDL and PQL	None	Report as estimated value ("J" flag)	None

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CCB = Continuing Calibration Blank
CCV = Continuing Calibration Verification
DoD = Department of Defense
ICB = Initial Calibration Blank
ICV = Initial Calibration Verification
LCS = Laboratory Control Sample
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit (Equivalent to LOD)
MS/MSD = Matrix Spike/Matrix Spike Duplicate
QC = Quality Control
PQL = Practical Quantitation Limit (Equivalent to LOQ)
QC = Quality Control
QSM = *Quality System Manual for Environmental Laboratories, Version 3, Final*
TOC = Total Organic Carbon

FIGURE

Authority / Lead Organization:
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Regulatory Review and Concurrence:
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Project Safety and Health Manager: Eric Wenger (816-822-3894)
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GeoCore Services, Inc
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Project Contact: Dale Robl (785-826-1616)

Environmental Priority Service, Inc.
Role: Direct Push and On-Site Analysis
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Role: Surveyor
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Burns & McDonnell
SINCE 1898

Figure 5-1
PROJECT ORGANIZATIONAL CHART
FORMER SCHILLING AIR FORCE BASE
SALINA, KANSAS

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APPENDIX A
ANALYTICAL MANAGEMENT LABORATORIES, INC.

2006 AML *Quality Assurance Plan*

2006 AML Standard Operating Procedures

AML Certifications

Performance Test Results

AML Statement of Laboratory Self Declaration of Compliance

AML Kansas Certification

(SUBMITTED ON CD)

2006 AML *Quality Assurance Plan*

QUALITY ASSURANCE PLAN

Analytical Management Laboratories

Analytical Management Laboratories

Quality Assurance Plan

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3.0 STATEMENT OF POLICY

Analytical Management Laboratories, Inc. (AML) is committed to providing quality data to its clients. AML will endeavor to ensure that analytical data generation activities performed by the laboratory will result in the production of data that are defensible, scientifically valid, of known quality, and can be used with a high degree of confidence by the user. AML intends to follow procedures referenced in this plan, standard operating procedures (SOPs) for field sampling and laboratory analytical activities that have been developed to supplement this plan, project specific documents such as quality assurance project plans (QAPP) developed to meet specific project requirements, and client contractual requirements. AML intends to conform to client specifications and regulatory agency guidelines for each project. The Quality Assurance (QA) program is intended to facilitate a sense of importance for quality control and quality assurance practices by managers, supervisors, analysts, and other participants in the data generation process.

The objectives of the QA program include but not limited to the following:

- Properly collect, preserve and store environmental samples.
- Maintain adequate chain of custody procedures from sample collection through reporting, data archiving and sample disposal.
- Use properly trained analysts to analyze samples within holding times using good laboratory and management procedures.
- Use approved methods, calibration procedures, precision and accuracy control limits to generate data of known quality.
- Document activities for verification, review, assessment, validation, and reporting of data.

The following sections of this QA plan detail the organizational structures and procedures through which laboratory results are generated.

4.0 ORGANIZATION AND RESPONSIBILITY

Analytical Management Laboratories, Inc. (AML) has laboratory facilities in and conducts field operations from its Olathe, KS office. The organizational chart for this facility is shown in Figure 4.1. The quality assurance responsibilities of the key personnel are as follows:

Board of Directors (BOD) is the corporate entity responsible for appointing company officers. Mr. Bill Said, Mr. Kendall Lindquist and Dr. Tenkasi Viswanathan serve as Directors.

Officers are members of an Executive Committee to deal with the day to day operations of the company. The Board of Directors has appointed three Officers to manage the operations of the company: President, Vice President/Treasurer and Secretary.

Executive Committee is responsible for oversight of the company operations. The company operations include the operation of the laboratory and it may include other activities of the company.

President is responsible for establishing corporate operations and the corporate policy. The President is also responsible for planning and overseeing the company and laboratory infrastructure, for negotiating contracts and for overall administration of the company. The president, by virtue of his position, is ultimately responsible and accountable for all the activities of the company and in establishing and maintaining good relationships with company clients. The President may provide guidance or delegate responsibility for some of these activities to the Vice President/Treasurer and or the Secretary. Mr. Bill said is the company President and he reports to the Executive Committee.

Vice President/Treasurer is responsible for assisting the President to engage in activities described for the President. The Vice President/Treasurer is also responsible for preparing financial reports, formulating personnel policies, review and restructuring of employee health and safety programs, and for the review of company assets and liabilities. The Vice President may provide guidance or delegate the responsibility for some of these activities to the President and or the Secretary. Mr. Tenkasi Viswanathan is the company Vice President/Treasurer and he reports to the Executive Committee.

Secretary is responsible for the day to day operations of the laboratory and to assist the President and Vice President/Treasurer in their activities. The Secretary is also responsible for general record keeping, maintaining personnel policies and benefits accounting, purchasing, maintenance of equipment, inventory and business records, and for employee health and safety programs. Mr. Kendall Lindquist is the Secretary and he reports to the Executive Committee.

Laboratory Director (LD) is a line position with responsibility for the overall operation of the laboratory. The Laboratory Director will have the following line responsibilities:

- Directing the overall laboratory program
- Developing and maintaining the sales and marketing plan for the laboratory program
- Recruitment and training of laboratory employees
- Identification of resources needed to complete laboratory tasks
- Establishing production priorities
- Reviewing laboratory procedures, practices and reports
- Performing client satisfaction surveys, and
- Guiding Project Managers and other laboratory staff

The Laboratory Director reports to the Executive Committee. Due to the relatively small size of the company, Mr. Bill Said also serves as the AML Laboratory Director.

Quality Assurance Director (QAD) is an independent line position reporting directly to the laboratory director. The QAD has sufficient authority and organizational freedom to identify problems; to initiate, recommend or provide solutions; and to verify implementation of solutions. The QAD is responsible for the following:

- Developing and updating a comprehensive quality assurance plan for the laboratory
- Serving as a QA liaison with clients and project managers
- Coordinating external accreditation and auditing activities
- Assessing the adequacy of quality control (QC) activities within the laboratory and keeping managers informed of their effectiveness
- Validating laboratory generated data with respect to established QC criteria
- Performing technical and system audits
- Preparing QA reports to management
- Coordinating the participation of the laboratory in external audit programs
- Communicating audit sample results to appropriate laboratory staff
- Coordinating corrective actions when needed and preparing corrective action reports to regulatory agencies
- Coordinating and/or assisting in the preparation and approval of Quality Assurance Project Plans (QAPPs)
- Preparation of Standard Operating Procedures (SOPs)
- Identifying staff training needs

The QAD works closely with the Laboratory Director and the Operations Manager to meet the objectives of the laboratory. Due to the relatively small size of the company, Dr. Tenkasi Viswanathan also serves as the QAD.

Operations Manager (OM) is a laboratory line position reporting directly to the laboratory director. The OM has sufficient authority to identify and task company resources to complete laboratory projects within schedule and budget and to implement the objectives of the laboratory and QA directors. The OM is responsible for the following:

- Implementing the AML quality assurance plan (QAP) or project-specific Quality Assurance Project Plans (QAPPs) for laboratory operations
- Serving as a point of contact with clients and/or AML project managers
- Supervising team leaders and/or analysts and technicians to complete laboratory analyses
- Interface with QAD in assessing and improving the adequacy of quality control (QC) activities within the laboratory
- Participating in the laboratory external audit programs
- Training laboratory staff
- Interface with QAD in coordinating corrective actions when needed
- Prepare laboratory reports and associated client invoices

The OM works closely with the Laboratory Director and the QAD to meet the objectives of the laboratory. Due to the relatively small size of the company, Mr. Kendall Lindquist also serves as the OM.

Project Managers are responsible for client service and overall project management. The duties of the project managers will include but not limited to the following:

- Establishing initial contact with client or prospect for the procurement of the project
- Determining the overall scope and profitability of the project
- Developing work plans, project schedule, and resource requirements

- Negotiating contracts in consultation with the LD, QAD, and OM
- Coordinating the efforts of Operations Manager, analytical team leaders, sample management, field sampling and data management crews for timely completion of the project
- Monitoring the progress of work and responding to client inquiries
- Reviewing and approving client reports and invoices
- Verifying that procedures and practices are appropriate for and consistent with client specifications and regulatory criteria
- Responding to post project inquiries

Project Managers report directly to the Operations Manager. The executive committee members and senior technical staff also serve as Project Managers for critical projects.

Team Leaders are responsible for supervising, organizing and directing work in their team. Team leaders report to the Operations Manager and interface with QAD and project managers on a routine basis to coordinate company efforts to complete projects to the satisfaction of clients. Team leaders have overall responsibility for technical operations assigned to their team. The duties of the team leaders include but not limited to the following:

- Ensuring effective maintenance, repair, and calibration of equipment and instrumentation assigned to their team
- Assuring adequate inventory of reagents and supplies
- Training employees as needed for proper performance of work by the team members
- Investigating and evaluating new methodology and equipment
- Assisting in development and implementation of quality control and quality assurance procedures, standard operating procedures, training manuals and other documents
- Making individuals in the team aware of relevant quality control practices
- Monitoring routine and non-routine aspects of the quality assurance plan as pertaining to their team and communicating with QAD about any situation requiring immediate attention
- Scheduling technical work for completion within required turnaround times
- Verifying that records are completed and maintained for each analysis conducted in their team.

Team Leaders report directly to the Operations Manager with the exception of the sales and marketing team, which reports directly to the Executive Committee. The executive and senior technical staff also serves as Team Leaders.

Team Responsibilities

Sales and Marketing Team is responsible for activities related to and including business development. This includes interfacing with laboratory director, prospects, potential clients, and current clients, project managers and QAO in procuring projects for the company. Responsibilities of this team include but not limited to the following: defining company services, developing brochures, mailing lists and other marketing materials, making sales calls in the assigned geographic area, conducting periodic market surveys, gathering information on competition, identifying new service areas, tracking government regulations and their impact on current service areas, identifying markets and demand for existing services, and client maintenance. This team is also responsible for organizing client parties, laboratory tours, and other activities aimed at promoting strong

relationships between the company and its clients. The sales and marketing team is currently managed by the Mr. Bill said, who also acts as the sales and marketing director.

Field Operations Team is responsible for all activities related to and including sample collection. This includes interfacing with clients, project managers and QAD in defining the scope of project field work, planning, work plan preparation, interfacing with subcontractors for field operations and sample collection. This team is also responsible for sample preservation, initiating chain of custody (COC), sample shipment sample pickup or sample delivery for laboratory analysis and for preparing necessary field reports. Mr. Robb Said currently manages the field operations team, which is limited to picking up samples from the field.

Mobile Laboratory Team is responsible for all activities related to and including sample analysis in the field. This includes interfacing with clients, project managers and QAD in defining the scope of project field laboratory work, planning, work plan preparation, interfacing with the field operations team or other contractors responsible for field operations. The duties of the mobile laboratory team may also include sample collection, which should be coordinated with the field operations team. This team is also responsible for sample preservation, initiating chain of custody, sample shipment or delivery for additional laboratory analysis and for preparing necessary field reports. The laboratory does not maintain a mobile laboratory at this time.

Sample Management Team is responsible for support activities related to sample collection and analysis. This includes the following: interfacing with clients, project managers, LIMS manager, and QAD in defining sampling supply requirements and providing supplies on timely basis. Supplies to be maintained include COC forms, coolers, glass and plastic containers, caps, labels, custody seals, preservatives, packaging materials and shipping supplies. This team is also responsible for maintaining chain of custody for samples received by the laboratory, sample and project log-in, and sample disposal. Additional duties include tracking, shipping or disposal of laboratory waste in accordance with regulatory requirements and preparation of glassware for laboratory analysis in accordance with standard operating procedures. Mr. Daniel Krugjohn currently manages the sample management team.

Organic Analysis Team is responsible for all activities related to and including sample analysis for organic parameters. This includes interfacing with clients, project managers, LIMS, and QAD in defining the scope of project laboratory work, planning and scheduling laboratory analysis. Analyses currently assigned to this team include but not limited to the following: volatile and semivolatile organic compounds, pesticides, PCBs, herbicides, and explosives analyses performed using gas chromatographs (GCs), high performance liquid chromatographs (HPLCs), gas chromatograph/mass spectrometers (GC/MS) and liquid chromatograph/mass spectrometers (LC/MS). Mr. Robert Hobson currently manages the organic analysis team.

Inorganic Analysis Team is responsible for all activities related to and including sample analysis for inorganic parameters. This includes interfacing with clients, project managers, LIMS, and QAD in defining the scope of project laboratory work, planning and scheduling laboratory analysis. Analyses currently assigned to this team include but not limited to the following: metals, minerals, demand, conventional waste, wastewater and drinking water characterization parameters; and analyses performed using atomic absorption spectrometers (AAs), inductively coupled plasma atomic emission spectrometers (ICP-AES), inductively coupled plasma/mass spectrometry (ICP/MS), ultraviolet, visible and infrared spectrometers (UV/VIS/IR), autoanalyzers and related equipment. Mr. Joseph Preheim currently manages the inorganic analysis team.

Data Management Team is responsible for support activities related to and including laboratory automation, data collection, data storage, data archiving, data retrieval, and report preparation. This

team is responsible for developing, implementing and training personnel in the use of state of the art computer hardware and software for proper laboratory data management including the laboratory information system (LIMS). Mr. Daniel Krugjohn currently manages the data management team.

Staff Chemists report to team leaders and they are responsible for completing duties assigned to them by their respective team leaders in a timely manner. Duties will include but not limited to the following: maintenance, repair and calibration of equipment assigned to them; extraction, digestion, and cleanup of sample extracts for instrumental analysis; instrumental analysis; reviewing own work and those performed by other chemists and technicians in accordance with standard operating procedures; completing checklists to document review; and making recommendations for technical decisions.

Staff Technicians coordinate their activities with staff chemists and they report to team leaders and they are responsible for completing duties assigned to them by their respective team leaders and/or staff chemists in a timely manner. Duties will include but not limited to the following: maintenance, repair and calibration of equipment assigned to them; extraction, digestion, and cleanup of sample extracts for instrumental analysis; sample analysis; and reviewing own work and those performed by other technicians in accordance with standard operating procedures; and making recommendations for technical decisions.

An individual may be assigned one or more responsibilities described above for optimum technical and financial operation of the company.

5.0 QUALITY ASSURANCE OBJECTIVES

AML has a comprehensive quality assurance program with the following specific objectives:

- To provide a controlled, traceable link through the entire determination process (sample collection through reporting);
- To provide a predetermined program for the acceptance or rejection of analytical data;
- To provide a system in which the laboratory can take and document action necessary to correct problems and insure the validity of reported laboratory data;
- To estimate the level of quality of each analytical system in a timely, efficient, and cost effective manner;
- To provide a system that is able to assist in early recognition of deficiencies, which might affect the quality of data; and
- To have in place a quality assurance audit program to insure that the plan, as established, is implemented and needed updates are made.

The QA Program is based on the following government documents:

1. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans (QAMS-005/80)".
2. Handbook for Analytical Quality Control in Water and Wastewater Laboratories (EPA, 1979).
3. Code of Federal Regulations (CFR), Title 40, Part 136.
4. EPA SW-846, "Test Methods for Evaluating Solid Waste".
5. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 2, June 2002.
6. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 3, March 2005.
7. US Army Corps of Engineers (USACE) Shell, EM 200-1-3, Appendix I.

The key to this program is adherence to the program during various phases of the project including: presampling discussions; sample container selection; sample collection, preservation, transportation and storage; sample login and tracking; laboratory analysis; data reduction, verification and reporting; and internal and external audit programs. These items are discussed in greater details in various sections of this manual and in the laboratory standard operating procedures (SOPs). The objectives of this program are summarized in this section.

5.1 QA Objectives for Data Measurement

The objective of data collection is to support the analytical programs of various clients and to produce data that is scientifically sound, legally defensible, and ultimately acceptable as

evidence in court. The statistical treatment of data by AML is generally in accordance with current scientific concepts included in standard references listed above.

Accuracy is defined as the closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.

Precision is a statistical term, which describes the closeness of agreement among a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by means of duplicate/replicate analyses for each analyte in a given matrix at concentrations above the MDL using a specified analytical method. Precision may involve the analysis of laboratory control samples (LCS) and/or matrix spikes (MS). Precision is normally expressed in statistical terms, such as the standard deviation of the values about their mean, or as the coefficient of variation (CV), which is the standard deviation of the mean expressed as a percentage of the mean. Precision is also expressed as relative percent difference (RPD) between duplicate measurements.

Bias is similarly a statistical parameter, which describes the systematic error as a deviation, with algebraic sign, of the mean value of many determinations of the analyte from its "true" value. This true value must either be known independently or determined by another independent method, which is known to have no bias itself.

The precision and accuracy of environmental sample analysis are greatly dependent on the sample matrix and the level of analyte concentration, both of which determine what values of precision and accuracy are acceptable for the intended use of analytical data. This dependence of precision and accuracy in analyses upon matrix and concentration requires the chemists involved to use professional judgment as to the range of the values acceptable for repeat determinations of the same sample. A list of all the individual analyses conducted by AML in all the possible matrices would be unmanageably large and of little practical value. Data generated within established concentration ranges for a particular analyte should meet pre-established quality control criteria for accuracy and precision.

Completeness and comparability of data are insured by adherence to a standard data set protocol and a checklist of required data deliverables for laboratory reports for each type of analysis conducted.

Representativeness is assured by employing a representative subsample for laboratory analysis from the environmental sample collected and submitted by the client. Each sample received by the laboratory is assigned a unique laboratory number identifying that sample. All work sheets will identify samples by that unique number. Work sheets will be recorded with dates and times and bear the laboratory analyst's initials. Each parameter data book should contain data and calculations associated with each independent determination. These include such things as sample weights or volumes, dilution factors, titration values, spectrometer readings, injection quantities, peak height or area readings from chromatograms, standard(s) identity and concentrations, as well as calculations related to each final value reported.

Laboratory data should be checked and initialed by the Analyst, Team Leader, and finally by the Operations Manager, QC/QA Manager/Director, or Laboratory Director to insure completeness of data and correctness of calculations in the report. For the occasional project involving very critical analyses on which serious action is contemplated, the QA Director will independently audit the entire data set for all analyses on the project sample(s). Work sheets, chromatograms, spectra, etc., associated with every analysis should be present with the parameter data book for the audit. These will give instrument operation parameters and details of instrument setup, such as columns used, mobile and stationary phases and concentrations, temperatures, detectors, spectrometer type, wavelength, etc., as applicable for the type of analysis.

5.2 Accuracy

The incorporation of laboratory control samples (LCS), standard reference materials (SRMs), matrix spike recovery studies, internal or external quality control (QC) check samples and other spiked samples in the normal analytical stream is the fundamental tool for monitoring the quality of a specific analytical process. AML compares the results of these samples to their true or assigned values to determine percent recovery, a measure of the accuracy for each set of analyses. The percent recovery is compared to acceptance ranges for evaluating associated sample data.

A minimum of one LCS will be analyzed with each set of samples, which is called a quality control batch (QC batch). For critical projects, LCS will be analyzed in duplicate. Warning and control limits for LCS are determined statistically from the mean and standard deviation of the observed recoveries as described elsewhere in the AML QAP document. LCS analyte recoveries within in-house control limits are acceptable without data qualifiers. Appropriate corrective action will be necessary for LCS recoveries outside the QC limits.

Ideally, each sampling and analysis project should establish its own acceptance criteria for LCS recoveries based on project data quality objectives (DQOs). Whenever available, the laboratory should use these project-specific limits in analytical reports. Project-specific limits may be incorporated by reference to administrative limits included in guidance documents such as the Department of Defense Quality Systems Manual (DoD QSM), USACE Shell document, etc. These administrative or default limits for LCS recoveries are based on the method quality objectives (MQOs) process, which are more general in nature. These administrative limits (e.g. DoD QSM, Appendix DoD-D, LCS Control Limits) may be used as default criteria when project specific limits are unavailable.

The in-house statistically derived control limits should be consistent with project specific limits or default limits. If the laboratory does not meet project specific requirements, efforts must be made to improve in-house method performance for the affected analytes. Negotiations with the client to use a different method, flag data, etc. may be necessary prior to sample analysis. When project-specified limits are wider than in-house limits, it is allowable to report data meeting project-specific limits but not the in-house limits. However, efforts should be made to investigate and correct the cause of failure based on the tighter in-house limits to minimize systematic errors.

Additionally, a matrix spike (MS) analysis on at least 5 % of the samples in each set (quality control batch) will be performed with a minimum of one spike per sample set. Matrix spike (MS) analysis is commonly performed in duplicate (MSD) and the frequency of MS analyses (MS and MSD combined) will amount to 10% for a batch of 20 samples. Required maximum QC batch size may be less than 20 samples for some analytical methods. Method requirements should be met in such cases. The spike recovery data for MS and MSD are obtained for each parameter of interest. A record of the recovery data of spiked analyses is kept for parameters analyzed by AML. The recovery data is recorded and presented on quality control charts to determine the acceptability of data for accuracy criteria. Quality control limits are determined either from statistical determinations and/or from administrative requirements such as the Department of Defense Quality Systems Manual (DoD QSM), USACE Shell document, etc. The administrative LCS recovery limits are also used in lieu of in-house statistical control limits for matrix spike samples.

5.3 Precision

The analysis of duplicate samples is the fundamental tool to measure the reproducibility of the analytical method, analyst, and/or the laboratory. Replicate analyses include sample replicates, LCS replicates or MS replicates. AML uses the results of these duplicate analyses to determine the precision of each analysis. Relative percent difference (RPD) is used to express precision between two replicate values. The precision data is compared to a pre-established criteria derived either from statistical sampling or administrative requirements such as the Department of Defense Quality Systems Manual (DoD QSM), USACE Shell document, etc.

AML analysts should conduct duplicate analyses on a minimum of 5 % of the samples in each set of samples analyzed. Alternatively, matrix spike recoveries should be performed in duplicate on a minimum of 5% of the samples in each set of samples analyzed, provided field samples have been collected for this type of analysis. Alternatively, LCS recoveries should be determined in duplicate for each set of samples analyzed. Maximum QC batch size may be less than 20 samples for some methods. Method requirements should be met in such cases. The results of these duplicate analyses are recorded on the RPD charts.

5.3 Comparability

AML's objective for comparability is to strive toward the comparability of sample parameters on similar matrices as they relate to precision and accuracy determinations. Strict adherence to QA/QC procedures, analytical methods, etc., promotes the comparability of one set of data to another or comparability of results performed at different times. AML will retain a copy of QA program manual in effect at the time analyses was performed for a reasonable period of time.

5.4 Representativeness

AML may not be involved in environmental sample collection activities. AML's objective for representativeness is to collect field samples and laboratory sub-samples that accurately depict the distinguishing characteristics of a sample source. When samples are collected by AML, sample collection procedures follow applicable AML Technical Guidance Documents to address representativeness. Representativeness is enhanced by collecting appropriate number of samples

necessary to characterize a site, by collecting and containerizing samples for VOCs analysis prior to the collection of less volatile parameters such as SVOCs and metals. Representativeness may also be enhanced by the collection of composite samples when applicable and by mixing samples to the extent possible in the field prior to aliquot removal. Whenever possible, representativeness of the analytical sample can be assured by thorough mixing (shaking, blending, or stirring) of the field sample prior to each weighing of an analytical sub-sample from the field sample. Results are considered reliable and representative if the sample distribution is within statistically defined bounds of the population mean and variance.

6.0 SAMPLING PROCEDURES

Sampling procedures are normally described in Sampling and Analysis Plans (SAP) or Field Sampling Plans (FSP), which are project specific documents. This chapter serves as a general guide to the sampling procedures that may be performed by Analytical Management Laboratories, Inc. (AML) for minor projects. Although the summary procedures described in this chapter are based on EPA guidance documents, a site specific Field Sampling Plan (FSP), which should incorporate text from AML technical guidance documents (TGDs) should be used to direct field sampling activities for major projects. The site specific FSP and associated AML TGDs will provide assurance that all preparations have been made in advance for the sampling crew to investigate the best possible procedures for any non-routine elements expected to be encountered. Procedures described in this section and/or AML TGDs are irrelevant when sampling is performed by the laboratory client.

Section 6.1 discusses the different types of samples, such as monitoring well samples, surface and industrial waters, soils, drums and debris. Section 6.2 states the minimum items and events that need to be documented during sampling to allow all interested parties to fully and accurately evaluate field and laboratory data. Section 6.3 includes some of the more common quality control measures that will be performed in the field, which will allow the samplers to demonstrate accuracy of the procedures performed. Section 6.4 relates to the proper selection and use of sample containers, preservatives and holding times. Combined, the information in these sections is intended to provide a well-documented quality operation regarding field sampling for the more common types of samples likely to be encountered.

AML and AML personnel are seldom responsible for sample collection. Therefore, a critical review of the sampling procedures described in this chapter, as part of a general review of the laboratory quality assurance plan (QAP) by external agencies is not recommended. The purpose of this chapter is to provide an overview of the sample collection process to the laboratory personnel involved in sample analysis. The intent of this chapter is simply to provide overall information related to the environmental data collection process.

6.1 SAMPLING SOURCES

6.1.1 Groundwater/ Monitoring Wells

Detailed procedures for sampling groundwater are described in the following EPA and USACE documents:

- Compendium of ERT Groundwater Sampling Procedures, EPA/540/P-91/007, January 1991
- RCRA Groundwater Monitoring: Draft Technical Guidance, EPA/530-R-93-001, November 1992
- US Army Corps of Engineers-Kansas City District, Standard Operating Procedure for Groundwater Low-Flow Purging, Revision 1.3, August 2002
- Engineering and Design – Monitoring Well Design, Installation, and Documentation at Hazardous Toxic, and Radioactive Waste Sites, EM 1110-1-4000, US Army Corps of Engineers, 1 November 1998.

These and other more recent documents are incorporated into this document by reference. The following is a summary of the groundwater sample collection procedures:

6.1.1.1 Initial Activities: All necessary equipment are assembled, calibrated and tested before sampling activities begin. For specific equipment types and models, calibration and maintenance procedures are provided in the manufacturer's manual. Between sampling locations, all items that come into contact with sample water are either disposed of, or thoroughly decontaminated using established procedures. Decontaminated or clean sampling equipment and samples should be kept out of direct sun, and laid out on plastic sheeting to minimize contamination. Initial inspection includes checking the well for aboveground damage, recording any evidence of damage to the well cap and lock in the field logbook, removal of the well cap and measurement of the headspace above the well for organic vapors with an organic vapor analyzer (OVA) or HNU analyzer. OVA readings are recorded in the field logbook and appropriate protective equipment are used in compliance with site the health and safety plan.

The depth to water and the depth of well are measured and recorded along with the time of measurement. The well measurements are made at an equilibrium level, at least 24 hours after any previous sampling of the well and before the wells are purged. If an electronic water level indicator is used, the following steps are followed:

- Check the alarm volume on the water level meter to ensure it is audible.

- Lower the probe into the well until the alarm is heard. Raise the probe until the alarm stops. Keep lowering and raising the probe until the point at which the alarm sounds if found. Then read the water level off the tape. Repeat measurement two more times.

- Finally, turn the volume off and lower the probe until the probe touches the bottom of the well. Record that depth. If there is slack in the line, a weighted cloth or steel tape should be used to obtain the overall depth measurement.

- After each of the above activities, record all pertinent information, including the following at a minimum:

    1. Depth to water
    2. Depth of well
    3. Well diameter
    4. Depth of water
    5. Calculated water volume
    6. Type of mechanism used to purge the well
    7. Mode of disposal of the purge water and information such as drum labels or other documentation, if purge water is collected.

The well is purged of three to five casing volumes of water by pumping or bailing. If the well does not recharge adequately, purging is complete upon bailing the well dry. The well is sampled immediately after purging. If this is not possible due to poor recharge rates, the well is sampled

within 24 hours after the completion of purging. Total purge-water volume is recorded in the field logbook.

6.1.1.2 Field Measurements; Because of the stagnant nature of the groundwater in the monitoring wells, it is necessary to purge the well casing of the water present. Well development should be performed in accordance with applicable federal, state and local regulatory requirements. Some of these requirements may specify that development continue until the readings for some indicator parameters like pH, conductivity, temperature, oxidation-reduction potential (ORP), dissolved oxygen (DO), or turbidity have stabilized; e.g., vary within a specified range. Stabilization is commonly considered to have been achieved after all parameters have stabilized for three successive readings. Generally three successive readings should be within ±0.2 for pH, ±3% for conductivity, ±10 mV for oxidation-reduction potential (ORP), ±1 degree Celsius for temperature, and ±10% for turbidity and DO. In general the order of stabilization is pH, temperature, and conductivity, followed by ORP, DO and turbidity.

The following equipments are used for field measurements on ground water samples:

- National Institute of Standards and Testing (NIST)- certified thermometer or temperature-measuring device calibrated in degrees Centigrade (C) with an accuracy of one-half degree C.
- pH meter with an accuracy of ± 0.1 pH units.
- Three pH buffer standards with pH values below and above the limits expected for the samples.
- Conductivity (specific conductance) meter, preferably with the capacity to report conductivity in micromhos/cm and a functional range of 0 to 50,000 micromhos/cm.
- Glass beaker for field measurement of pH, conductivity, and temperature.

A clean glass beaker is filled with water from the well for field measurements of pH, specific conductivity, and temperature. The field measurements are made as follows:

1. Immediately measure the temperature of the water to the nearest one-half degree C with a NIST mercury thermometer or temperature-measuring device. Record in field logbook.
2. Calibrate specific conductivity meter according to manufacture's recommendations. Adjust the specific conductivity meter for the temperature of the water sample. Obtain three readings of specific conductivity for each well, using separate samples, and record each of the specific conductivity readings in the field logbook to the nearest micromhos/cm.
3. Calibrate the pH meter according to the manufacturer's recommendations. Obtain three readings of pH for each well using separate samples for each reading. Rinse probe with deionized water between readings. Record the values in the field logbook.
4. Make any additional entries in the field logbook as required such as the presence of an oily phase.

5. Dispose of samples and decontamination fluids with purge water form the well.

Calibration and maintenance procedures for each piece of equipment will be according to the manufacturer's requirements. An equipment maintenance log is kept to record the life history of each measuring and testing device used in activities affecting quality. The following information is recorded:

- Equipment identification (name) and control number.
- Date of calibration or maintenance.
- Condition of equipment.
- Activity performed on instrument (calibration or maintenance).
- Adjustments made an accuracy of equipment before and following calibration (where applicable).
- Record of equipment failure or inability to meet specifications (where applicable).
- Identification of person performing calibration/maintenance.
- Next due date for calibration or maintenance.

As part of normal field operations, some instruments may require calibration before and during field use. This field operation calibration is kept separate from pre-field-use calibrations. Field calibrations are recorded in field logbooks as part of the normal field data collection process.

No specific operation or field calibration instructions are included for the pH meter or specific conductivity meter because specific make and model of each instrument used may vary from project to project. These instruments are operated and calibrated in accordance with the manufacturer's instruction manuals accompanying the equipment.

Low flow pumping is an appropriate purging and sample collection mechanism, whenever there is a desire to limit the quantity of investigation derived waste and to collect groundwater samples with minimal disturbance to the formation. Pumping rate is adjusted until the recharge rate equals it and drawdown is minimal (< 1'). Flow-through cells can be used to measure pH, temperature and specific conductance. Flow-through cells are also excellent devices to measure dissolved oxygen (DO), oxidation-reduction potential (ORP) and other parameters that are air sensitive.

6.1.1.3 Groundwater Sampling Equipment Selection and Use: Two categories of ground water sampling devices are used; grab samplers and positive displacement (submersible) pumps. Whenever possible, sampling equipment is dedicated to a particular well. The following general guidelines regarding the selection of appropriate sampling equipment are used: (a) Sampling equipment should be constructed of inert material; (b) Sample collection equipment should not alter analyte concentrations, cause loss of analyte via sorption, or cause gain of analytes via desorption, degradation, or corrosion; (c) Sampling equipment should be designed such that materials such as Viton, Tygon, silicone, or neoprene components do not come into contact with the ground water sample since these materials have been demonstrated to cause sorptive losses of contaminates; (d) Sampling equipment should cause minimal sample agitation and should be selected to

reduce/eliminate sample contact with the atmosphere during sample transfer; and (e) Sampling equipment should not allow volatilization or aeration of samples to the extent that analyte concentrations are altered.

6.1.1.4 Collecting Groundwater Samples: Whenever possible, monitoring well sampling are scheduled to progress from the well that is expected to be least contaminated to the well that is expected to be most contaminated, to minimize the potential for cross contamination of samples that may result from inadequate decontamination of sampling equipment. Samples are collected and containerized according to the volatility of the target analytes. The preferred collection order for some of the more common ground water analytes is as follows:

1. Volatile organics (VOA or VOCs) and total organic halogens (TOX);
2. Dissolved gases and total organic carbon (TOC);
3. Semivolatile organics (BNAs, or SVOCs);
4. Metals and cyanide;
5. Major water quality cations and anions;

Other procedures to consider include the following: filling the sample bottles directly from the bailer or pump; carefully pouring the contents of the bailer into the prepared bottles, to minimize the possibility of aeration; holding the sample bottles over a bucket to collect any spillage during filling; not allowing the bailer and rope to touch the sample bottle; and filling the bottles for VOC analysis without any headspace first and capping them tightly. The following procedures also apply to the use and operation of ground-water sampling equipment:

(a) Check valves should be designed and inspected to ensure that fouling problems do not reduce delivery capabilities or result in aeration of samples.

(b) Sampling equipment should never be dropped into the well, as this will cause degassing of the water upon impact.

(c) Contents of the sampling device should be transferred to sample containers in a controlled manner that will minimize sample agitation and aeration.

(d) Decontaminated sampling equipment should not be allowed to come into contact with the ground or other contaminated surfaces prior to insertion into the well.

(e) Ground water samples should be collected as soon as possible after the well is purged. Water that has remained in the well casing for more than about 2 hours has been demonstrated to exchange gases with the atmosphere and to interact with the well casing material.

(f) The rate at which a well is sampled should not exceed the rate at which the well was purged. Ideally, the rate of sample collection should be approximately the same as the actual ground water flow rate. Because this is typically not possible, low sampling rates, approximately 0.1 L/min., are suggested. Low sampling rates will help to ensure that

particulates, immobile in the subsurface under ambient conditions, are not entrained in the sample and that volatile compounds are not stripped from the sample. Pumps should be positioned at or near the screen interval and operated at rates less than 0.1 L/minute when collecting samples for volatile organics analysis.

(g) Pump lines should be cleared at a rate of 0.1 L/min. or less before collecting samples for volatiles analysis so that the samples collected will not be from the period of time when the pump was operating more rapidly.

(h) Pumps should be operated in a continuous, non-pulsating manner so that they do not produce samples that are aerated in the return tube or upon discharge.

(i) Ground water samples collected for analysis for organic constituents or parameters should not be filtered in the field.

6.1.1.5 Decontamination: **N**on-dedicated sampling equipment is thoroughly decontaminated between sampling points to prevent carryover. Decontamination is not required for duplicate samples collected from the same monitoring well. Equipment to be decontaminated is disassembled if possible and washed with a detergent solution (such as Alconox) and scrubbers. The equipment is thoroughly rinsed with potable water followed by distilled water. The rinsate water are collected and analyzed in the laboratory for proper disposal. The water can be poured onto the nearby ground if written permission is obtained from the client.

Field measurement equipment, pH probe, etc., are decontaminated using procedures recommended by the manufacturer. Generally these also require distilled water rinses and wipes. Equipment (probes, beaker, etc.,) used to measure the water level, pH, specific conductivity, and temperature of water from the well are decontaminated between each sampling event by rinsing with deionized water. The following cleaning procedure is used for decontaminating sampling equipment when organic constituents are of interest:

(a) Wash the equipment with a non-phosphate detergent.
(b) Rinse the equipment with tap water.
(c) Rinse the equipment with isopropanol.
(d) Rinse the equipment with organic-free water.

If acetone, hexane, or methanol is not analytes of interest, methanol, hexane and acetone in that order may be chosen instead of isopropanol. The following cleaning procedure is used for decontaminating sampling equipment when inorganic constituents are of interest:

(a) Wash the equipment with a non-phosphate detergent.
(b) Rinse the equipment with tap water.
(c) Rinse the equipment with dilute (0.1N) hydrochloric or nitric acid.
(d) Rinse the equipment with reagent water.

Dilute hydrochloric acid is preferred over nitric acid when cleaning stainless steel because nitric acid may oxidize the steel. In all cases, waste decontamination fluids are containerized until the investigators determine (e.g. through analytical testing) whether fluids should be treated or disposed of as a hazardous waste. Following decontamination, equipment is allowed to dry thoroughly in a dust-free environment. If the equipment is not to be used again immediately, they are packaged and properly stored to protect it from dust and dirt. Equipment may be wrapped in aluminum foil (shiny side on the outside) and placed in a plastic bag. A label is affixed to the outside wrapping summarizing the decontamination procedure and stating the date of decontamination. Decontaminated sampling equipment is never placed on the ground or on other contaminated surfaces prior to insertion in the well.

6.1.2 SURFACE WATER

Detailed procedures for sampling surface water are described in the EPA documents, "Compendium of ERT Surface Water and Sediment Sampling Procedures", EPA/540/P-91/005, January 1991" and "Handbook for Sampling and Sample Preservation of Water and Wastewater", EPA-600/4-82-029, September 1982, which are incorporated into this document by reference. The following is a summary of the procedures:

The surface water is sampled by submerging a clean beaker or other suitable container into the water, filling it with minimal agitation, then transferring the contents into a clean bottle containing appropriate preservatives. Overfilling the sample container is avoided since this may cause the preservative to become diluted and spill outside the container. The outside of the containers are then wiped to protect personnel from possible exposure during transport, analysis and to minimize contact with secondary materials such as the insides of coolers. Care is taken to minimize sediment agitation, since this may change the make-up of the sample and possibly the chemistry and level of contamination.

Any observations concerning homogeneity and degree of agitation are noted in the field logbook. Particular attention is also needed concerning health and safety issues. Very large flows, difficult access, unstable footing, etc. are potential hazards. Vapors coming from the water or from nearby discharge pipes can be hazardous. The safety situation is assessed and assistance sought, if necessary, from the health and safety officer.

A wide variety of sampling equipment is available for surface water sampling. Some equipment is automated and dedicated to collect aliquots at time intervals or representative of flow velocity. The manufacturer's instructions are followed for installation, operation and decontamination. The following considerations are employed in the selection of proper sampling equipment: (a) Sampling equipment should be constructed of inert material; (b) Sample collection equipment should not alter analyte concentrations, cause loss of analyte via sorption, or cause gain of analytes via desorption, degradation, or corrosion; (c) Sampling equipment should be designed such that Viton, Tygon, silicone, or neoprene components should not come into contact with the water sample. These materials have been demonstrated to cause sorptive losses of contaminates; (d) Sampling equipment should cause minimal sample agitation and should be selected to reduce/eliminate sample contact with

the atmosphere during sample transfer; and (e) Sampling equipment should not allow volatilization or aeration of samples to the extent that analyte concentrations are altered.

Samples are collected an containerized according to the volatility of the target analytes. The preferred collection order for some of the more common analytes is as follows:

1. Volatile organics (VOA or VOCs) and total organic halogens (TOX);
2. Dissolved gases and total organic carbon (TOC);
3. Semivolatile organics (BNAs, or SVOCs);
4. Metals and cyanide;
5. Major water quality cations and anions;

For equipment re-used at another sample location, decontamination is performed to minimize carry-over. The decontamination procedures are identical to the procedures described for groundwater sampling (section 6.1.1.5).

6.1.3 SOIL SAMPLES

Detailed procedures for sampling soil and sediment samples are described in the EPA documents, "Compendium of ERT Surface Water and Sediment Sampling Procedures", EPA/540/P-91/005, January 1991" and "Compendium of ERT Soil Sampling and Surface Geophysics Procedures", EPA OSWER Directive 9360.4-02, PB91-921273, January 1991, which are incorporated into this document by reference. The following is a summary of the procedures:

Soil samples can be collected in a variety of ways. Borings from drilling equipment, such as hollow stem augers or Geoprobes, can be placed directly into the appropriate container. The soil sample container for volatiles is filled first, and then the other containers are filled in order of decreasing volatility. Because of the widely varying concentration in soils and the adhering nature of clay soils, the decontamination of the sampling equipment is very important. The decontamination follows the general procedures listed in Section 6.1.1.5. Rinsate blanks are collected to check for the adequacy of the decontamination procedures. This consists of pouring distilled water over the decontaminated sampling equipment immediately following a routine environmental sampling event and collecting the rinse-water for laboratory analysis. Contamination in the rinsate blank may indicate problems related to the decontamination procedures, and the potential for false positives in the analytical data.

Soil samples collected for the analysis of volatile organic compounds may require sample collection and analysis in accordance with the newer SW-846 Method 5035. Samples for this method should be collected in accordance with SOPs developed to address issues as precise weight measurements and options for field preservation of samples.

6.1.4 TAP SAMPLES

Water samples collected from taps require purging just like monitoring wells since contaminants can concentrate in the water, and be retained inside the pipes if the water has not been flowing. Prior to sampling any aeration devices are removed and the tap turned on to maximum for 30 seconds or

longer for longer pipe lengths. Samples are collected directly into sample containers with appropriate preservatives, making sure to avoid overfilling.

The following considerations are employed in the selection of proper sampling equipment: (a) Sampling equipment should be constructed of inert material; (b) Sample collection equipment should not alter analyte concentrations, cause loss of analyte via sorption, or cause gain of analytes via desorption, degradation, or corrosion; (c) Sampling equipment should be designed such that Viton, Tygon, silicone, or neoprene components known to do cause sorptive losses of contaminates do not come into contact with the water sample; (d) Sampling equipment should cause minimal sample agitation and should be selected to reduce/eliminate sample contact with the atmosphere during sample transfer; and (e) Sampling equipment should not allow volatilization or aeration of samples to the extent that analyte concentrations are altered.

Samples are collected an containerized according to the volatility of the target analytes. The preferred collection order for some of the more common tap water analytes is as follows:

1. Volatile organics (VOA or VOCs) and total organic halogens (TOX);
2. Dissolved gases and total organic carbon (TOC);
3. Semivolatile organics (BNAs, or SVOCs);
4. Metals and cyanide;
5. Major water quality cations and anions;

6.1.5 SEWERS/OUTFALLS/DISCHARGE PIPES

Detailed procedures for wastewater are described in the EPA documents, "Compendium of ERT Surface Water and Sediment Sampling Procedures", EPA/540/P-91/005, January 1991" and "Handbook for Sampling and Sample Preservation of Water and Wastewater", EPA-600/4-82-029, September 1982, which are incorporated into this document by reference. The following is a summary of the procedures:

The sampling of these water sample sources follows surface water and tap sampling (sections 6.1.3 and 6.1.4) closely. In most cases it will be necessary to obtain flow measurements before sample collection. The following considerations are employed in the selection of proper sampling equipment: (a) Sampling equipment should be constructed of inert material; (b) Sample collection equipment should not alter analyte concentrations, cause loss of analyte via sorption, or cause gain of analytes via desorption, degradation, or corrosion; (c) Sampling equipment should be designed such that Viton, Tygon, silicone, or neoprene components known to do cause sorptive losses of contaminates do not come into contact with the water sample; (d) Sampling equipment should cause minimal sample agitation and should be selected to reduce/eliminate sample contact with the atmosphere during sample transfer; and (e) Sampling equipment should not allow volatilization or aeration of samples to the extent that analyte concentrations are altered.

Samples are collected an containerized according to the volatility of the target analytes. The preferred collection order for some of the more common wastewater analytes is as follows:

1. Volatile organics (VOA or VOCs) and total organic halogens (TOX);
2. Dissolved gases and total organic carbon (TOC);
3. Semivolatile organics (BNAs, or SVOCs);
4. Metals and cyanide;
5. Major water quality cations and anions;

6.1.6 DRUMS AND TANKS

Detailed procedures for sampling waste including drummed waste are described in the EPA documents, "Compendium of ERT Waste Sampling Procedures", EPA/540/P-91/008, January 1991" and "Waste Analysis at Facilities that generate, treat, store and dispose of hazardous wastes: A Guidance Manual", OSWER 99384.4-03, April 1994, which are incorporated into this document by reference. The following is a summary of the procedures:

The sampling of drums and tanks is potentially one of the most dangerous among the sampling activities performed. The health and safety officer and the owner of the drums/tanks are consulted before proceeding with sampling. The level of safety required is the first determination needed prior to pre-sampling activities such as drum inspection, drum staging and drum opening. Generally, a small volume of sample is sufficient for most laboratory analysis. Equipment used for opening drums include bung wrench, drum deheader, hand pick, pickax, backhoe spike, hand spike, and other hydraulic or pneumatic devices. Equipment used for sampling include glass thief tubes, composite liquid waste samplers (COLIWASA), Bacon Bomb Samplers, Sludge Judges, bailers and subsurface grab samplers.

Selection of proper sampling equipment, collection and containerizing samples are performed according to the guidelines described for groundwater sample collection (section 6.1.1).

6.1.7 BULK (CHIP, WASTE PILE, SWEEP)

Detailed procedures for sampling bulk samples, which include construction materials, chips, sweeps, wastepiles and other bulk samples are described in the EPA document, "Compendium of ERT Waste Sampling Procedures", EPA/540/P-91/008, January 1991", which is incorporated into this document by reference. The following is a summary of the procedures:

Chip sampling, which is appropriate for porous surfaces, is accomplished with a hammer and chisel or an electric hammer. Sweep sampling, which is appropriate for the collection of dust, is accomplished using a dedicated brush to sweep material into a dedicated dustpan. Waste pile solids are sampled with stainless steel scoops, shovels, trowels, spoons, stainless steel hand augers, post hole augers, bucket augers, grain samplers or sampling triers. Bulk samples such as construction debris can be difficult to sample. Generally a portion of the material of interest is sampled and placed in an airtight, inert container. Clean aluminum foil may be the only practical material for certain type of samples, which are used for non-volatile analyses and many inorganic tests.

6.1.8 WIPES

Detailed procedures for performing wipe sampling are described in the EPA document, "Compendium of ERT Waste Sampling Procedures", EPA/540/P-91/008, January 1991", which is incorporated into this document by reference. The following is a summary of the procedures:

Wipe samples are collected from smooth surfaces to check for surficial contamination. The sample location is measured (usually 100 square centimeters) and marked. A gauze pad is soaked in a suitable solvent (such as hexane) and the area is wiped vertically and then horizontally to ensure complete coverage. The pad is then placed in a screw top, septum lined vial for laboratory analysis.

6.2 DOCUMENTATION OF FIELD ACTIVITIES

6.2.1 FIELD LOGBOOK

The following field information is documented in the field logbook:

* Well identification
* Well depth
* Static water level depth and measurement technique
* Presence and thickness of immiscible layers and detection method
* Well yield (high or low) and well recovery after purging (slow, fast)
* Well purging procedure and equipment
* Purge volume and pumping rate
* Time well purged
* Collection method for immiscible layers
* Sample withdrawal procedure and equipment
* Date and time of collection
* Well sampling sequence
* Types of sample bottles used and sample identification numbers
* Preservatives used
* Parameters requested for analysis
* Field observation of sampling event
* Name of collector
* Weather conditions, including air temperature
* Internal temperature of field and shipping containers.
* Information pertaining to field activities is recorded on a daily basis. (e.g.: a record of all personnel, including subcontractors and visitors to the site).
* List of equipment and standards used for the sampling, including equipment identification numbers and expiration dates of standards.

6.2.2 SAMPLE LABELS

Labels are attached to each sample container to prevent sample misidentification. Sample labels are sufficiently durable to remain legible even when wet and contain, at a minimum, the following information:

* Sample identification number
* Name and initials of the collector
* Date and time of collection
* Place of collection
* Parameters requested (if space permits).
* Preservatives employed

The samples are labeled by recording the above information directly on the sample containers. Alternatively, the sample collector may use multiple-part labels consisting of a unique identification number that is placed on the container. At least two copies of the descriptive information for the samples (referenced to the identification number) are made. One copy is kept in a separate file or logbook, and a second copy is shipped with the samples to the laboratory.

6.2.3 SAMPLE CUSTODY SEALS

In cases where sample leave the sample collector's immediate control (e.g. for shipment to the laboratory), a custody seal is placed on the shipping container or on the individual sample bottles. Custody seals provide prevention or easy detection of sample tampering. The custody seal bears the signature of the collector and the date signed. The custody seal can be placed on the front and back of a cooler, around the opening of a polyethylene overpack bag or on the lid of each sample container. Caution is exercised in doing any of the above since experience has shown that the seal may not always adhere to plastic coolers, and that the coolers may arrive at the destination without the appropriate seal. Taping over custody seals on plastic coolers with a transparent tape may allow removing tape and the custody seal without disturbing the custody seal. Custody seals on sample containers may become wet from melting ice or condensation and fall off. Use of a better adhesive is recommended for custody seals that are applied to the containers.

6.2.4. CHAIN OF CUSTODY RECORD

The tracing of sample possession is accomplished by use of a chain-of-custody record as described in Chapter One of SW-846. Multiple-copy chain-of-custody record forms may be used to assure each person in the chain of custody link gets a copy of this document. At a minimum, the chain-of-custody record should contain the following information:

* Sample number
* Signature of collector
* Date and time of collection
* Sample type (e.g. ground water)
* Identification of sampling point (well).
* Number of containers
* Analyses requested
* Preservatives used
* Signature of persons involved in the chain of possession
* Inclusive dates and times of possession
* Internal temperature of shipping container at the time of sample shipping
* Internal temperature of container when opened at the lab
* Remarks section to identify potential hazards or to relay other information to the lab.

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6.2.5 SAMPLE ANALYSIS REQUEST SHEET

The sample analysis request sheet should accompany the samples on delivery to the lab and clearly identify which sample containers have been designated for each requested parameter. The sample analysis request sheet may be included in or be a part of the chain-of-custody record. The addition of preservatives should be noted on the sample analysis request sheet. The sample analysis request sheet should include the following information:

* Name of person shipping/receiving the sample
* Name and address of the analytical laboratory
* Lab sample number (if different from field number)
* Date of sample receipt
* Analyses requested
* Internal temperature of shipping container upon opening in the lab
* Preservatives added in the field.

6.3 FIELD QUALITY CONTROL MEASURES

Field quality control (QC) samples are collected in the same manner as regular investigation samples with regards to sample volume, containers, and preservation. The concentrations of any contaminants found in blank samples are used to assess contamination pathways and to qualify usability and not for correcting the sample data. The contaminant concentrations in blanks are documented, and if the concentrations are greater than the applicable regulatory level and/or greater than the field sample results, resampling may be performed.

The following field QC guidelines are appropriate:

- **Field Replicates or QC Splits** - one per batch per matrix or every 20 samples, whichever is more frequent. The field replicates may be submitted to the laboratory as any other environmental (blind replicate) sample without identifiers marking it as a field replicate. The identity of the original and the replicate samples may not be disclosed to the laboratory (double blind replicates).

- **Matrix Spikes (MS) and Matrix Spike Duplicates (MSD) -** One set of MS/MSD per matrix or every 20 samples, whichever is more frequent. Identify the sample to be collected in triplicate in the FSP for normal, MS and MSD analyses. The laboratory may use LCS/LCSD as a substitute for MS/MSD when project samples are not accompanied by MS/MSD samples, which may be the case for some sample delivery groups. An alternate solution in the laboratory would be to batch samples without an accompanying MS/MSD pair with samples containing a MS/MSD pair, if it is permissible.

- **QA Splits** - 10% of all samples collected for analysis by an independent laboratory.

- **Rinsate Blanks** - One per batch per matrix or every 20 samples, whichever is more frequent. This sample is obtained by pouring distilled or deionized water through or over decontaminated sampling equipment after an environmental sample was collected. This will test for the thoroughness of the decontamination procedures.

- **Field Blanks** - One per batch per matrix or every 20 samples, whichever is more frequent. This sample is collected by exposing the distilled water used for decontamination to field conditions and pouring them into a regular sample container. This sample checks for field-introduced contamination from the atmosphere, distilled water, sample containers or sampling equipment. Field blanks may be skipped if rinsate samples are collected.

- **Trip Blanks** - One per sample shipment cooler containing samples for volatiles analysis. This sample is generated by the laboratory using organic free water. The water is placed in a VOA vial and transported to and from the site without ever being opened. One trip blank should be placed in every cooler used to store or transport samples for VOCs analysis. Trip blanks may also be used for petroleum volatiles such as gasoline range organics (GRO), natural gases such as methane, ethane and ethene (MEE) and volatile pesticide compounds (EDB, etc.). This QC sample is used to check for possible cross contamination within the cooler from accompanying high level samples or environmental conditions.

6.4 CONTAINERS, PRESERVATIVES AND HOLDING TIMES

Clean sample containers or vials are sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. If certified containers are not used, the cleanliness of a batch of precleaned bottles is verified in the laboratory, prior to sampling in the field.

Samples for the analysis of volatile organic compounds are collected with no headspace in the containers. Immediately after samples designated for volatile organics analysis have been filled and capped, they are checked for headspace. In most cases, the entire sample may be emptied from the container and the container refilled if headspace is noted in the sample. The container should not be "topped off" to fill the additional headspace. If headspace is observed after samples have been collected, field logs and laboratory analysis reports should note the headspace, if present, in the sample containers at the time of receipt by the laboratory, as well as at the time the samples were first transferred to the sample containers.

Splitting samples is a common practice. Normally, aliquots from the sampling device are alternately emptied into each container receiving a split until the containers are full. When splitting samples for volatile organics compound analysis (VOA), each VOA vial is completely filled and sealed. Vials are not kept open while the sample is distributed between vials.

It is important to correctly match sample matrix and analyte with the proper containers and preservatives. Details on sample containers are described in the following documents:

- Table 2-36 (Page 2-48 of Volume 1A) of “Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/Chemical Methods, Revision 3, December 1996

- Code of Federal Regulations (CFR), Title 40, Part 136.3(e)
- US Army Corps of Engineers, EM 200-1-3, Appendix B, February 1, 2001

Information contained in these EPA documents is incorporated into applicable AML SOPs and in this document by reference. In general, organic samples are collected in glass containers and inorganics samples are collected in plastics. Although preservatives are required for some parameters, they are often added to prolong the holding time allowance and to improve analytical results. Adjustment to the size and number of sample containers may be made in the field (e.g. wells do not produce enough water) or the laboratory (e.g. one sample container may be shared between a number of tests, e.g. SVOCs, pesticides, and DRO) to adjust for sample availability and to minimize sample disposal.

7.0 SAMPLE CUSTODY

A sample is defined as physical evidence collected from a facility, site or the environment. The primary objective of sample custody is to provide accurate, verified, and traceable records of sample possession and handling from sample container shipment through laboratory receipt and sample disposition.

7.1 Custody Definitions

Sample Custody is defined as records and documentation that is required to trace a sample from point of origin through disposal after analysis. Chain-of-Custody (COC) is a formal written record of all events associated with custody of a specific sample. A sample is in custody if the following are satisfied:

- It is in the field investigator's or the transferee's actual possession; or
- It is in the field investigator's or the transferee's view, after being in his/her physical possession; or
- It was in the field investigator's or the transferee's physical possession and then he/she secured it to prevent tampering; or
- It is placed in a designated secure area.

7.2 Custody Documentation Overview

The flow of documented information on a sample received in the laboratory includes the following: sample labels (provided with sample containers), custody seals (as needed), chain-of-custody records, sample log-in records, internal sample custody records, and sample disposal logs. For some projects, this definition may include sample preparation logs, sample run logs, sample analysis logs, client reports, etc.

7.2.1 Sample Containers

7.2.1.2 Appropriate containers should be used for sample collection. Samples should be preserved in the field as described in the applicable method. A list of sample containers for common parameters, preservatives and holding times are shown in Figure 7.1. This list has been reproduced from Appendix B of the USACE document, EM-200-1-3.

7.2.1.2 A lesser number of containers may be collected for soil samples, if analyses for multiple parameters (e.g. SVOCs, pesticides, PCBs, metals, etc.) are needed on the same sample. Separate aliquots are needed for each of the following analyses: matrix spikes (MS), matrix spike duplicates (MSD), or matrix duplicates (MD). Although the laboratory can provide appropriate containers and preservatives, it is the responsibility of the sample collector to ensure that requirements included in the applicable field sampling plan (FSP) are met.

7.2.2 Sample Receipt and Acceptance

7.2.2.1 Samples received in the laboratory are initially handled and processed by the sample custodian or his/her designee. A COC record form originating from either AML or the client will accompany most samples received by the laboratory. An example of the AML COC form is provided in Figure 7.2. An example of the sample labels and cooler custody seals used by AML are shown in Figure 7.3.

7.2.2.2 If requested, the COC will start when pre-cleaned containers are shipped to the field from the laboratory and the sample custodian will sign the COC form as the sample containers are relinquished to the client. For most samples, COC begins from the time

samples are collected by either AML or client personnel. For samples received without a COC record, the laboratory will originate COC from the time of receipt. This form must be filled out completely and signed in the "relinquished by" section by the person delivering the samples.

7.2.2.3 Upon receipt of samples, AML Sample Custodian will inspect the package of samples and document these observations on a checklist (Figure 7.4). Items checked normally include the following: temperature of the temperature blank or the cooler (for details, see AML SOP on temperature measurements and calibration of temperature measurement devices), type of coolant used, pH of the samples except VOCs analysis samples (for details, see AML SOP on Sample Login procedure and the AML SOP on pH measurements for accurate pH measurements, when required), presence/absence of custody seals, integrity of containers, packing material used, quantity of samples, and the presence/absence of air bubbles in aqueous samples requiring analysis for VOCs. After ensuring that the information about samples and the COC agree, the sample custodian will sign the COC and document the date and time received. Any discrepancies will be noted on the checklist. A portable document file (pdf) copy of the checklist will be created and emailed to clients that require this documentation. The client will also be contacted by telephone if immediate client response is needed to address serious deficiencies.

7.2.2.4 Samples received in one shipment will be assigned an AML work order number, which is similar to the sample delivery group (SDG) number used by EPA. The AML work order number will be used to track the group of samples received at one time, which may require similar processing/reporting. Multiple AML work order numbers may be assigned for a single shipment of samples if the number of samples exceed 20 or the samples require different type of reports (e.g. Level III report for some samples and Level IV reports for other samples) and/or turnaround times (TAT) such as 7-day TAT for some samples and the standard TAT for others. The original COC form, traffic records and any accompanying field information documentation will be kept in the project data files. Laboratory sample numbers will be assigned to each sample received on the original COC. All entries on the COC record must be signed in ink or typewritten. Any errors should be corrected by drawing a single line through the error, signing and dating the correction. A pdf copy of the completed COC and/or the log-in report will be emailed to the client as an acknowledgment of receipt of samples to clients that require this documentation. The client will also be contacted by telephone if immediate client response is needed to address serious deficiencies.

7.2.2.5 AML policy dictates that all samples sent to the laboratory for analysis are accepted by the laboratory and assigned a unique number for tracking purposes. Specific samples may not be acceptable if improper containers (e.g. wide-mouthed 2-Oz jars for VOCs in water, and plastic containers for organochlorine pesticides) are used for sample collection or the samples are not collected properly (e.g. air bubbles/head space for aqueous VOCs samples). If samples are received at the lab in broken or leaking containers, improperly preserved, requiring analytical testing not performed by the lab, etc., the samples may be logged into the system and the client or project manager is contacted before any analysis begins. Any problems of this nature should be documented on the COC form, the checklist. The corrective action taken by the laboratory through client discussions will also be documented in project reports. Data qualification for improperly collected samples will remain the responsibility of client.

7.2.2 Sample Login

Once the chain-of-custody record has been completed and signed, a copy of the COC form is made and is used as a sample log-in and tracking form. The samples are then logged into the laboratory information management system (LIMS). This is the responsibility of the sample custodian. The information to be tracked includes the following:

- Date/Time of sample receipt
- Courier used by the sample shipper including shipping records, if any
- Name and signature of person receiving the samples (sample custodian)
- AML work order number
- Sample Delivery Group (SDG) and Case Number for EPA projects
- Client Project number
- Client Project name
- Client and AML Sample numbers
- Date/Time of sample collection
- Customer sample identifiers from COC sheet
- Sample matrix
- Due date - based on holding times or client request whichever is sooner.
- Tests required including the sample preparation and analysis methods
- Condition of the shipping container
- Condition of the sample bottles
- Appropriate preservation of samples (to be checked by the analyst for VOCs)
- Presence or absence of traffic reports
- Verification of agreement or non-agreement of information on shipping documents.
- Sample tag identification numbers cross-referenced to the client sample numbers
- Presence/absence of custody seals on shipping container(s)
- Discrepancies, if any

7.2.3 Sample numbering and identification

7.2.3.1 During the log-in procedure, each sample is assigned a unique number, which is used exclusively to identify and track samples within the laboratory and to generate reports using the LIMS. Samples designated by the client for MS and MSD analyses are marked with these requests on the comments section for each test. The MS and MSD samples are assigned unique AML sample numbers for each test during sample preparation with a link to the original sample that was used for the unspiked sample analysis.

7.2.3.2 Samples requiring analysis for a single parameter such as VOCs in soil or water will have multiple containers. Whenever a sample consists of more than one container, each container is identified with the same sample number with an appended letter code to identify the specific container that may be used to perform analysis. This letter code is not a part of the AML sample number since it may make it difficult to extract sample data from the LIMS database. The letter codes should only be used in sample extraction and/or analysis logs to indicate the source of specific sample. When sample heterogeneity is suspected by the sample custodian/analyst, the containers collected for sample, MS and MSD may be combined and homogenized (not allowed for VOCs and related analyses) before aliquots are transferred to the original sample containers.

7.2.3.3 Samples requiring multiple parameters (e.g. SVOCs, pesticides, and metals in soil) may be able to share the sample contained in a single 4-Oz jar. However, soil samples for tests such as explosives, nitrocellulose, and nitroglycerin that requires preliminary sample handling steps such as air drying and homogenization prior to sample extraction should be kept separate after the preliminary sample handling step.

7.2.3.4 The LIMS Manager and/or sample custodian is responsible for assigning sample numbers. At this time two digit suffixes (01 to 99) are added to the AML work order number (four digits) to assign sample numbers for each sample received and logged as one group. An example LIMS report is shown in Figure 7.5.

7.2.4 Sample Preservation

The laboratory provides containers and preservatives. The preparation of these preservatives is documented in a logbook. When the laboratory purchases pre-cleaned containers with preservatives, the documentation provided by the vendor is used. The preservation of samples will be checked and additional preservatives will be added to samples requiring them (e.g. nitric acid to groundwater samples with pH>2). The solid samples requiring VOCs analyses by SW-846 Method 5035 will be inspected in accordance with applicable AML SOP. The sample weights, type of preservatives used, etc. will be documented as shown in Figure 7.6.

7.2.5 Sample Storage

After log-in is completed the sample custodian, in designated areas stores the samples, until analytical processing begins and the appropriate analytical sections are notified of sample receipt. These areas are:

Purgeable Organics (VOCs - Water)	=	Water Samples stored in a dedicated upright refrigerator located in the VOA laboratory.
Purgeable Organics (VOCs - Soil)	=	Soil Samples preserved with acid or methanol are stored in the same dedicated upright refrigerator used for storing water samples located in the VOA laboratory.
Sample extracts for VOA	=	Dedicated, upright VOA refrigerator
Purgeable Organics (VOCs - Soil)	=	Frozen soil Samples for 5035 analysis are stored in a dedicated upright freezer located in the VOA laboratory.
Semivolatile organics	=	Cold storage area
Other organics	=	Cold storage area
Metals and inorganics	=	Cold storage area
pH	=	Not stored whenever possible; taken to the analyst for immediate analysis
Sample extracts (non-VOA)	=	Upright refrigerator
Metals digestates	=	Dry sample storage area

To ensure that samples and extracts are properly stored, the sample custodian records the storage location on the chain-of-custody login form. The storage location is also written on the sample container to ensure that the samples are returned to the proper location after analysis.

7.2.5.1 Cold Storage: The cold storage area is a large walk-in refrigerator that is kept at approximately 4°C ± 2 ^{o}C. The cold storage area is coded and equipped with a certified thermometer to track the temperature of its contents. It is located in the shipping and receiving area.

7.2.5.2 Upright Refrigerator: Several upright refrigerators are located in the general chemistry lab. Each refrigerator is coded and equipped with a certified thermometer to track the temperature of its contents. It is used to store samples, standards, extracts or digestates. These refrigerators are kept at 4°C.

7.2.5.3 Freezer: The freezer for storing frozen 5035 soil samples is coded and equipped with a certified thermometer to track the temperature of its contents. The freezer is kept at –12°C or lower.

7.2.5.3 Dry Storage Area: Storage room in laboratory used to store samples and digestates that do no require refrigeration.

7.2.5.4 High Concentration Sample Storage: High concentration samples are stored in sealed containers and jars in an isolated cabinet dedicated to this purpose.

7.2.5.5 An internal chain of custody document (see Figure 7.7) is also generated by the LIMS for projects requiring chain of custody documentation within the laboratory.

7.2.6 Sample Preparation

Sample extraction and digestion are documented by the preparation technician on sample extraction or digestion logs designed for each type of analysis. These are described in appropriate method specific AML standard operating procedures. Analyst's name or initials are included on all sample run and analysis logs. The LIMS is designed to prepare a summary for each sample preparation batch (Figure 7.8). The LIMS assigns unique AML sample numbers to the common QC elements such as MB, LCS, LCSD, MS and MSD. The LIMS also assigns unique AML sample numbers to QC elements that are unique to some analyses such as SD and PDS samples.

7.2.7 Sample Analysis

Sample analysis is documented in sample analysis logs specific to each instrument or analysis type. Run logbooks are also kept to record activity of each major instrument. These are described in appropriate method specific AML standard operating procedures. Analyst's name or initials are included on all sample run and analysis logs. The LIMS is designed to prepare a summary for each sample analysis batch (Figure 7.9). The LIMS assigns unique AML sample numbers to common QC elements such as CCVs and CCBs.

7.2.8 Sample Disposal

Determining the proper sample disposal procedure is the responsibility of the Project Manager. Disposal requirements are documented on a sample disposal record (see Figure 7.10). After all analyses are complete, the sample disposal record is given to the sample custodian who will dispose of the sample as described on the sample disposal record. The sample disposal record is then filed in a three-ring notebook. Waste disposal procedures, which include sample disposal practices, are included in specific SOPs.

7.3 Sample and Laboratory Security

Sample and laboratory security is maintained by keeping all outside access doors, except the front door, locked at all times. The sample custodian is on duty to keep vigil on the walk-in cooler. Visitors are required to register at the front desk and are escorted by laboratory personnel while in the facility.

7.4 Inter-laboratory Custody

If it is necessary to transfer samples/extracts to another laboratory, a completed, signed chain-of-custody record should be included with the transfer. If sample extracts are being transferred to another lab, it is also necessary to attach a copy of the preparation log and related documents.

8.0 ANALYTICAL PROCEDURES

AML employs EPA/State/Other Regulatory Agency approved methodology for laboratory and field analysis of samples, when required by regulation. The ultimate responsibility for analytical method selection lies with the client, who may choose regulatory agency approved methods or alternative methods. In those cases where no specific soil or sediment method exists, AML will use its standard operating procedure, which may be closely related to an approved water method.

8.1 Analytical Methods

Analytical results provided by AML are used in site investigations, for compliance with RCRA, NPDES, UST or SDWA regulations, groundwater monitoring, for determining the progress of environmental remediation and other specialized use. The methods used are those specified by the U.S. Environmental Protection Agency (EPA), other federal agencies, state agencies, and other organizations. Methods used in the laboratory are recognized, published methods. AML maintains a library of most common methods for easy access to such information. AML also maintains standard operating procedures (SOPs), which are based on one more of the applicable methods. A list of AML SOPs are shown in Tables 8.1 through 8.12

8.2 Reagent and Chemical Storage

The storage procedure for all reagents and chemicals used in the laboratory is described in Table 8.13. All chemical containers are clearly labeled with the date of receipt and the expiration date (if applicable). Expired chemicals and reagents are not used for laboratory analysis.

Table 8.1
Quality Control, Quality Assurance (QC/QA)
AML Administration and Management SOPs

SOP Number	SOP Title
Q-Audit	Internal Quality Assurance (QA) Audits and Performance Evaluation
Q-Complaint	Complaint Resolution
Q-ControlCharts	Generation and Maintenance of Control Charts
Q-Dataverification	Data Reduction, Review, Verification, and Reporting Procedures
Q-EDD	Electronic Data Deliverables
Q-Ethics	Ethics Statement
Q-ManualIntegration	Manual Integration - AML Policy and Procedures
Q-MDL-RL-MQL	IDL, MDL, LLR, RL, PQL, and Initial Demonstration of Performance (IDP)
Q-ProjectMgmt	Project Management
Q-Rounding	Significant Figures
Q-Software	Software Acquisition, Validation and Training
Q-SOPs	Preparation, Distribution, and Control of Standard Operating Procedure (SOP) Documents
Q-Training	Technical Training

Table 8.2
Laboratory Infrastructure
AML QC/QA SOPs

SOP Number	SOP Title
Q-Cleaning	Glassware Cleaning Procedures
Q-DataMgmt	Data Storage, Retrieval, and Management Procedures
Q-Gravimetric	Gravimetric Measurements and Control of Balances and other Weight Measurement Devices
Q-logbooks	Lab Notebooks Control, Use, Review, and Approval
Q-SampleMgmt	Sample Receiving, Log-in, Storage, Tracking, and Management Procedures
Q-Sonicator	Sonicator Tuning
Q-Standards	Analytical Reagents and Standards
Q-Subsampling	Laboratory Subsampling from Sample Containers
Q-TemperatureControl	Temperature Measurements and Control of Ovens, Coolers, Refrigerators, and Freezers
Q-Volumetric	Volumetric Measurements and Control of Volume Measurement Devices
Q-WasteMgmt	Waste Management and Disposal Including Sample Disposal
Q-Water	Reagent Water

Table 8.3
Gas Chromatography/Mass Spectrometry (GC/MS)
AML Organic Analysis SOPs

SOP Number	SOP Title
O-Acids-Alcohols-GCMS	SPME Extraction and GC/MS Analysis of Alcohols and Volatile Organic Acids
O-BNA-SVOCs	Base Neutral and Acid (BNA) Semivolatile organic compounds (SVOCs) by GC/MS
O-DW-SOCs	Safe Drinking Water Act (SDWA) Semivolatile Organic Compounds (SOCs) by Solid Phase Extraction (SPE) and Instrumental Analysis by GC/MS (EPA 525.2)
O-5035	Soil sample collection and preservation procedures for the analysis of VOCs by SW-846 Method 5035
O-FieldandQALab Guide-5035	Guidance for Field and QA Lab Personnel for Handling Soil Samples Collected Using AML 5035 SOP
O-PAH-GCMS-SIM	Polynuclear Aromatic Hydrocarbons (PAHs or PNAs) by GC/MS in the Selected Ion Monitoring (SIM) Mode (EPA 625 and SW-846 method 8270C)
O-PCDDs-PCDFs-LRMS	Polynuclear Dibenzo-p-Dioxins (PCDDs) and Polynuclear Dibenzofurans (PCDFs) by High Resolution Gas Chromatography/Low Resolution Mass Spectrometry (HRGC/LRMS), EPA 613 and SW846-8280A
O-SoilVOCs-5035	Soil Sample Collection and Preservation Procedures for the Analysis of VOCs by SW-846 Method 5035
O-VOCs-GCMS	Volatile Organic Compounds in water and Soil by GC/MS (EPA Methods)

Table 8.4
Gas Chromatographic (GC) Analysis using FID and TCD Detectors
AML Organic Analysis SOPs

SOP Number	SOP Title
O-TPHDiesel	Total petroleum hydrocarbons (TPH) - Diesel fraction (DRO) by GC/FID (EPA SW-846 8015B mod. And OA-2)
O-TPHGasoline	Total petroleum hydrocarbons (TPH) Gasoline fraction (GRO) by GC/FID (EPA SW-846 8015B mod. And OA-1)
O-MEE	Analysis of Methane, ethane, ethene (MEE) in Aqueous Samples by Headspace Sampling and GC/FID (EPA SW-846 Method 8015B mod. And EPA RSK-175)
O-Hydrogen	Hydrogen Analysis by GC/TCD

Table 8.5
Gas Chromatographic (GC) Analysis using ECD and NPD Detectors
AML Organic Analysis SOPs

SOP Number	SOP Title
O-EDB&DBCP	EDB and DBCP by Microextraction and GC/ECD Analysis (EPA Methods 504, 504.1, and SW-846 8011)
O-Herbicides	Chlorophenoxy herbicides by GC/ECD (EPA 515.1 and SW-846 8151)
O-OCPesticides	Organochlorine pesticides by GC/ECD (EPA Methods 508.1, 608, and SW-846 8081A)
O-PCBs	Polychlorinated biphenyls (PCBs) by GC/ECD (EPA 508, 608, and SW-846 8082)

Table 8.6
High Performance Liquid Chromatography (HPLC)
AML Organic Analysis SOPs

SOP Number	SOP Title
O-Explosives	Niroaromatics, nitramines, Metabolites of Explosives and related compounds (Nitroglycerin, PETN, and Nitroguanidine) by HPLC (SW-846 8330 and 8332)
O-PAH-HPLC	Polynuclear aromatic hydrocarbons (PAHs or PNAs) by HPLC (EPA 550.1 and SW-846 8310)

Table 8.7
Elemental Analysis
AML Inorganic Analysis SOPs

SOP Number	SOP Title
I-ICP-AES-Metals	Metals analysis by inductively coupled plasma - atomic emission spectroscopy (ICP/AES), EPA 200.7 and SW-846 6010B
I-ICPMS-Metals	Metals analysis by inductively coupled plasma - mass spectrometry (ICP/MS), EPA 200.8 and SW-846 6020A
I-GFAA-Metals	Metals analysis by graphite furnace atomic absorption spectroscopy (GFAA), EPA 200 Series, EPA 200.9 and SW-846 7000 Series
I-Hardness	Hardness (SM 2340B)
I-CEC	Cation exchange capacity (CEC) of soils by sodium acetate treatment and ICP/AES analysis (SW-846 9081)
I-CVAA-Mercury	Mercury in soil and water by cold vapor atomic absorption (CVAA) Technique, EPA 245.1, 245.5, SW-846 7470A and 7471A
I-3010A-Prep	Acid digestion of aqueous samples for total metals analysis by flame atomic absorption spectroscopy (FLAA) or ICP/AES
I-3020A-Prep	Acid digestion of aqueous samples for total metals analysis by GFAA or ICP/MS
I-3050B-Prep	Acid digestion of soil, sediment, and sludge samples for total recoverable metals analysis by FLAA, GFAA, ICP/AES or ICP/MS
I-Cr-VI-Water	Hexavalent chromium analysis in aqueous samples by manual spectrophotometry (SW-846 7196)
I-Cr-VI-Soil	Hexavalent chromium analysis in soil, sediment, and waste samples by alkaline digestion (SW-846 3060) and manual spectrophotometry (SW-846 7196)

Table 8.8
Ion Chromatography
AML Inorganic/Organic Analysis SOPs

SOP Number	SOP Title
I-Anions-IC	Determination of inorganic anions by Ion Chromatography (IC), EPA 300.0, 300.1, and SW-846 9056
I-GN-9056	Determination of Guanidinium Nitrate by Ion Chromatography (IC), EPA 300.0 and SW-846 9056
I-Perchlorate	Determination of perchlorate by Ion Chromatography (EPA 314.0 and SW-846 9058)

Table 8.9
Waste Testing
AML SOPs

SOP Number	SOP Title
W-Ignitability	Ignitability (SW-846 Chapter 7)
W-Flashpoint-CC	Ignitability - Flashpoint Determination by closed cup tester (SW-846 1010, 1020A and ASTM D-3278-78)
W-Corrosivity-pH	Electrometric Measurement of pH in Soil and Waste
W-Corrosivity-Steel	Corrosivity to Steel Determination (SW-846 1110)
W-Reactivity	Reactivity - cyanide and sulfide (SW-846 Chapter 7.3)
W-PaintFilter	Paint filter liquids test (SW-846 9095A)
W-TCLP	Toxicity chracteristic leaching procedure (TCLP), SW-846 1311A
W-SPLP	Synthetic precipitation leaching procedure (SPLP), SW-846 1312

Table 8.10
Demand and Nutrients
AML Inorganic Analysis SOPs

SOP Number	SOP Title
	Demand
I-BOD	Biological Oxygen Demand (BOD), EPA 405.1 and SM 5210B
I-COD-RA	Chemical Oxygen Demand (COD) by Manual Spectrophotometry (EPA 410.4, Hach 8000, and SM 5220)
I-Oil&Grease-TPH-1664	Oil and grease, gravimetric
I-Phenolics	Phenolics by Spectrophotometry (EPA 420.1, SM 5530, and SW-846 9065)
I-Surfactants	Surfactants or Methylene Blue Active Substances (MBAS) by Spectrophotometry, EPA 425.1
I-TOC-RA	Total Organic Carbon (TOC) in Soil and Water (EPA 415, SW-846 9060, and SM 5310)
	Nutrients
I-Ammonia-350.2	Ammonia by Distillation and Colorimetry (EPA 350.2)
I-Ammonia-ISE	Potentiometric Measurement of Ammonia (EPA 350.3 and SM 4500-NH_3-D)
I-Nitrate-Nitrite-353-354	Nitrate and Nitrite by Manual Cadmium Reduction and Spectrophotometry (EPA 353.3, 354.1, SM 4500-NO_2^-, and SM 4500-NO_3^-)
I-TKN-351.3	Total Kjeldahl Nitrogen (TKN), EPA 351.3 and SM 4500-N_{org}
I-OrganicNitrogen	Organic Nitrogen (EPA 350.2, 350.3, 351.2, 351.4, and SM 4500-N_{org})
I-Phosphorus-365.3	Phosphorus by Spectrophotometry (EPA 365.3 and SM 4500-P)

Table 8.11
Solids and Physical Properties
AML Inorganic Analysis SOPs

SOP Number	SOP Title
	Solids
I-TDS-160.1	Total Dissolved Solids (TDS) or Residue, filterable, gravimetric, EPA 160.1
I-TSS-160.2	Total Suspended Solids (TSS) or Residue, non-filterable, gravimetric, EPA 160.2
I-Solids-160.3	Total Solids or Residual, total, gravimetric or total, EPA 160.3
I-VolatileSolids-160.4	Volatile Solids, or Residue, volatile, gravimetric, EPA 160.4 and SM 2540E
I-Settleable-Solids-160.5	Settleable Solids or Residue, settleable matter, volumetric, Imhoff cone, EPA 160.5 and SM 2540F
	Physical Properties
I-Acidity	Acidity by Titrimetry (EPA 305.1 and SM 2310B)
I-Alkalinity	Alkalinity by Titrimetry (EPA 310.1)
I-pH-water	Electrometric Measurement of pH in Water (EPA 150.1, SM 4500, SW-846 9040B)
I-Conductance	Specific Conductivity (EPA 120.1, SM 2510, and SW-846 9050A)
I-Temperature	Temperature (EPA 170.1 and SM 2550)
I-Color	Color by ADMI Filter method (EPA 110.1 and SM 2120E)
I-Turbidity	Turbidity (EPA 180.1)

Table 8.12
Miscellaneous Tests
AML Inorganic Analysis SOPs

SOP Number	SOP Title
I-Chloride-325.3	Chloride by Titrimetry (EPA 325.3, SM 4500-Cl^-, and SW-846 9077)
I-Chloride-9253	Chloride by Argentometric Titrimetry (SM 4500-Cl^- and SW-846 9077/9253)
I-Chlorine	Chlorine, total residual by titrimetry (DPD-AES), EPA 330.4 and SM 4500-Cl
I-Cyanide-Total	Cyanide, amenable to chlorination by spectrophotometry, EPA 335.1 and 9014
I-Cyanide-Amenable	Cyanide, total by spectrophotometry, EPA 335.2 and SW-846 9014
I-DO	Oxygen, dissolved by membrane electrode, EPA 360.1
I-NC	Nitrocellulose analysis by spectrophotometry
I-Sulfate	Sulfate, turbidimetric, EPA 375.4
I-Sulfide	Sulfide, colorimetric/titrimetry, EPA 376.1, 376.2, SM 4500 and SW-846 9034

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Table 8.13

REAGENT and CHEMICAL STORAGE

Chemical Type	Method of Storage
Acids	Stored by acid type, in original containers in vented cabinets designed for acid storage.
Bases	Stored in original containers in vented cabinet designed for storage of bases
Organic Solvents	Stored in original containers in vented cabinets designed for storage of organic solvents. These cabinets are located in the organic extraction room.
Organic solvents (Flammable)	Stored in original containers in fire cabinets, located in the explosion-proof organic extraction area.
Dry Chemicals	Stored in cabinets located in the chemical storage room. Chemical are separated according to hazard code (e.g. oxidizers, corrosives, health hazards).

9.0 CALIBRATION PROCEDURES AND FREQUENCY

9.1 Laboratory Equipment

Analytical Management Laboratories is equipped with state-of-the-art instrumentation to provide quality analytical data to clients. A list of the instrumentation maintained by Analytical Management Laboratories is shown in Table 9.1. Information contained in this table is dynamic and items on this table are subject to constant change.

9.2 Standard Receipt and Traceability

Standards are purchased from commercial sources in mixes designed for the specific methods or as neat compounds. Certificates of analysis are shipped with each ampule by the vendor. The standards are certified to meet or exceed the criteria established by the USEPA.

Upon receipt, the standard materials are dated and assigned AML tracking numbers. Standard logbooks are maintained in different sections of the laboratory to document traceability of working standards back to neat materials or prepared stock mixes. Standards are assigned a lot number that provides unique identifiers, which are documented in laboratory notebooks along with date of preparation, initials of the analyst, concentration, expiration date, and solvent (if applicable). A standard preparation narrative is included in this section to detailing the preparation steps for each standard.

9.3 Standard Sources and Preparation

AML maintains an inventory of materials to produce stock standards or purchases of stock standards from commercial vendors. Laboratory preparation of all lab-prepared stock, intermediate, and working standards is documented by the responsible analysts. Tables 9.2 and 9.3 present standard sources and preparation protocols for various sections of the laboratory. Information contained in this table is dynamic and items on this table are subject to constant change. Field instruments requiring calibration standards (conductivity meters and pH meters) use the same sources as laboratory instrumentation.

9.4 Laboratory Instrument Calibration

Summary of the calibration procedures followed at AML shown below should meet or exceed EPA method requirements. AML has also incorporated USACE guidance (EM 200-1-3, Appendix I) for most methods and EPA SW-846 Method 8000C guidance for the calibration of chromatographic instruments. USACE guidance involves an initial calibration verification using a second source standard immediately after initial calibration. It also involves maximum %RSDs for initial calibration and maximum %D for calibration verification. See applicable method specific AML SOPs for more details on calibration.

9.4.1 Metals

9.4.1.1 Inductively Coupled Plasma (ICP) Atomic Emission Spectrometers (AES)

The inductively coupled plasma atomic emission spectrophotometer (ICP-AES) is standardized daily with one or more single concentration standard solutions containing metals of interest and a blank. After standardization, the calibration standard is analyzed immediately as a sample (AML improvement). The observed result should be within ±5% of the true value and the RSD for replicate measurements should be within 5%. The dynamic linear range is verified by analyzing a high level standard (HLSTD) at or near the upper limit of the linear range (USACE improvement). The observed result should be within

±10% of the true value and the RSD for replicate measurements should be within 5% for HLSTD. The initial calibration verification (ICV) and initial calibration blank (ICB) standards are analyzed. The observed result for ICV should be within ±10% of the true value and the RSD for replicate measurements should be within 5%. Quality control criteria for acceptance of ICB depend on the analytical program such as EPA contract laboratory program (CLP). The ICB result should be less than one half of the aqueous method quantitation limit (MQL), which is used as the default criteria at AML. The MQL is verified daily by analyzing a low-level stock solution (CRI) containing metals of interest at 2 times the CLP contract required detection limit (CRDL) for most analytes, which serves as the AML method quantitation limit (MQL). The observed result for CRI should be within ±20% of the true value.

The MQL is defined as the concentration level at which the measured value is within 20% of the true or expected value. If the 20% criteria cannot be consistently met at twice the CRDL, the concentration of the MQL is increased until compliance is attained. In principle, the upper limit of calibration is defined by the instrument linear range, which is established annually. In practice, however, the upper limit of calibration is verified by running a high standard (HLSTD) after instrument standardization.

Additionally, initial calibration is followed by the analysis of interference check standards A and AB (ICSA and ICSAB). The recovery of target analytes should be within ±20% for ICSAB to be acceptable. Continuing calibration verification (CCV) and blank (CCB) standards are run before and after every 10 samples. Sample data are bracketed by calibration verification standards that are within QC limits (same as ICV and ICB) for data to be acceptable.

Additional interference check samples, check samples to verify linear range, etc. are run at analyst's discretion. The AML standard operating procedure for ICP analysis contains additional details.

9.4.1.2 Inductively Coupled Plasma (ICP) Mass Spectrometers (MS)

The inductively coupled plasma mass spectrophotometer (ICP-MS) is standardized daily using a procedure that is very similar to that described for ICP-AES. After calibration, a high level standard (HLSTD), a method quantitation limit check sample (CRI or MQL), an initial calibration verification sample (ICV) and an initial calibration blank (ICB) standards are analyzed. Quality control criteria for acceptance of ICV, ICB, HLSTD, and CRI are similar to those described for ICP-AES analysis. This is followed by the analysis of interference check standards (ICSA and ICSAB) which should be within QC limits. The interference check samples are not analyzed at the end of an analytical batch for ICP-MS analysis. Continuing calibration verification (CCV) and blank (CCB) standards are run after every 10 samples and sample data are bracketed by calibration verification standards that are within QC limits for data to be acceptable. For sample data to be acceptable, the internal standard recoveries should be within the 30% to 130% range. The AML standard operating procedure for ICP-MS analysis contains additional details.

9.4.1.3 Atomic Absorption (AA) Spectrometer

Furnace atomic absorption spectrophotometers are calibrated daily with a minimum of three standards and a blank. Cold vapor mercury analyzers that employ atomic absorption are calibrated with a minimum of five standards and a blank. An initial calibration verification standard (ICV) and an initial calibration blank (ICB) are analyzed immediately upon calibration, which should meet method criteria

that are included in the AML standard operating procedures for GF (GFAA) and cold vapor (CVAA) atomic absorption analysis. Continuing calibration verification (CCV) and continuing calibration blanks (CCB) standards analyzed after every 10 samples should have recoveries within QC limits for bracketed data to be acceptable. AML SOP on mercury analysis and AA measurements should be used for additional guidance.

9.4.2 General Chemistry

9.4.2.1 Ion Chromatograph

Ion chromatographs are calibrated with a minimum of five calibration standard solutions and a calibration blank. Response factor method or linear regression (preferred) may be employed for initial calibration (ICAL). If non-linear regression (quadratic curve) is used, additional standards (six or seven) are analyzed. Before analysis of samples, a second-source initial calibration verification (ICV) standard and an initial calibration blank are analyzed. The second-source ICV standard may be replaced by a second-source laboratory control sample (LCS). Continuing calibration verification standards (CCVs) are analyzed immediately before the analysis of samples and after every 10 samples. The correlation coefficient (r) for linear regression must be greater than or equal to 0.995 for ICAL to be acceptable. When the coefficient of variation (r^2 or COD) is used to measure the goodness of the fit for linear or non-linear regression, they must be greater than or equal to 0.990. QC limits for CCVs are specified on AML SOPs dealing with specific (e.g. perchlorate, anions, guanidinium nitrate, etc.) ion chromatographic analysis procedure. The calibration verification standards should be within QC limits for associated data to be acceptable.

9.4.2.2 Ultraviolet and Visible (UV-VIS) Spectrophotometers

The spectrophotometer is calibrated initially with calibration standards at multiple concentration levels (three to five depending on test parameter). Linear regression is used to generate calibration curves. The correlation coefficient (r) for linear regression must be greater than or equal to 0.995 for ICAL to be acceptable. When the coefficient of variation (r^2 or COD) is used to measure the goodness of the fit for linear or non-linear regression, they must be greater than or equal to 0.990. Calibration verification standards are analyzed immediately following the ICAL and after every 10 samples. The calibration verification standards must be within the QC limit for associated data to be acceptable. Calibration procedures should be as specified on specific AML SOP dealing with the specific parameter for analysis.

9.4.2.3 Infrared (IR) Spectrophotometer

AML does not currently offer IR methods for analysis. When applicable, the infrared spectrophotometer should be calibrated initially with a minimum of five standards. The correlation coefficient (r) for linear regression must be greater than or equal to 0.995 for ICAL to be acceptable. When the coefficient of variation (r^2 or COD) is used to measure the goodness of the fit for linear or non-linear regression, they must be greater than or equal to 0.990. A calibration verification standard is analyzed immediately upon calibration, and after every 10 samples. The calibration verification standards must be within the QC limits for associated data to be acceptable. Calibration procedures should be as specified on specific AML SOP dealing with the specific parameter for analysis.

9.4.2.4 Conductivity Meter

The cell constant of each meter is determined at a minimum annually by the analysis of five KCl standards. To verify the cell constant, a second-source initial verification standard (ICV) is analyzed at the beginning of each working day, using a KCl standard in the expected range of sample concentrations. A calibration verification standard (CCV) is analyzed immediately upon calibration, and after every 10 samples. The measured values for ICV and CCV standards should be within 10% of the true or expected values. For meters not having automatic temperature compensation, all samples are analyzed at 25°±2° C. AML SOP on conductivity measurements should be used for additional guidance.

9.4.2.5 pH Meter

The pH meter is calibrated daily with two standard buffers at pH 7.0 and either 4.0 or 10.0, and checked with a third buffer at 10.0 or 4.0 which should indicate pH within ±0.10 pH units of its given value. A calibration verification standard is analyzed immediately upon calibration and after every 10 samples. The calibration verification standards must be within the default control limits (± 10%) for associated data to be acceptable. Manual or automatic temperature compensation is performed depending on the meter. AML SOP on pH measurements should be used for additional guidance.

9.4.2.6 Total Organic Carbon (TOC)

Although the instrument can be operated over a wide concentration range (1- to 6000- mg/L), the instrument is standardized for the most sensitive range (1 to 20-mg/L) in accordance with manufacturer instructions. Initial calibration for sample analysis is performed using a calibration blank and three to five standards using four replicates for aqueous samples. A smaller number of replicates may be used for soil sample analysis. The correlation coefficient (r) for linear regression must be greater than or equal to 0.995 for ICAL to be acceptable. When the coefficient of variation (r^2 or COD) is used to measure the goodness of the fit for linear or non-linear regression, they must be greater than or equal to 0.990. A second source initial calibration verification standard (ICV) is analyzed immediately upon initial calibration (ICAL). Continuing calibration verification standards (CCV) are run before analysis of samples and after every 10 samples. The calibration verification standards must be within the QC limits (±10%) for associated data to be acceptable. For details on calibration procedures and guidance on TOC measurements AML SOP on this parameter should be used.

9.4.2.7 Ion Selective Electrodes (ISE)

Ion selective electrodes are calibrated with a minimum of five standards. Linear regression applied to a plot of the log of the standard concentrations versus potential should result in a correlation coefficient equal to or greater than 0.995. Calibration verification standards are analyzed immediately upon calibration and after every 10 samples. The calibration verification standards must be within the default control limit criteria (± 10%) for associated data to be acceptable.

9.4.2.8 Total Organic Halogen (TOX)

AML does not currently offer TOX analysis. When applicable, the instrument should be standardized in accordance with manufacturer instructions and calibrated as described in SW-846 Method 9020B section 7.2. Initial calibration involves checking the adsorption efficiency of the newly prepared batch of carbon

using adsorption efficiency standards in duplicate. The net recovery should be within ± 10% the true value. Initial calibration also involves verifying the pyrolysis procedure in duplicate with instrument-calibration standards. The net response should be within ± 10% the true value. Initial calibration is verified (ICV) in duplicate with a second source standard, which should be within ± 10% the true value. Calibration is verified (CCV) in duplicate before and after the analysis of each group of ten pyrolysis samples. The net response should be within ± 10% the true value.

9.4.2.9 Bomb Calorimeter

AML does not currently offer this analysis. When applicable, the energy equivalent of the bomb calorimeter is determined quarterly by analyzing six standard benzoic acid tablets. A fuel oil standard is analyzed in duplicate for every batch of samples. The results of this sample should be within the established or default control limits criteria (±20%) for associated sample data to be acceptable.

9.4.2.10 Dissolved Oxygen (DO) Meter

DO meters are calibrated prior to use either by Winkler titration or the air calibration technique. Calibration is verified initially before sample analysis (ICV) with continuing verification (CCV) after every ten samples. The observed value should be within ± 10% the true value. AML SOP on dissolved oxygen measurements should be used for additional guidance.

9.4.2.11 Temperature

All laboratory and field thermometers are calibrated annually by comparison with an NIST-certified thermometer. Field meters with automated temperature compensation are checked before use with a calibrated thermometer. The observed temperature should be within ± 1° the true value. Otherwise, appropriate corrections should be applied. AML SOP on temperature measurements should be used for additional guidance.

9.4.3 Gas Chromatographs

9.4.3.1 GC Volatiles

Since a number EPA methods exist under this general category, AML SOP dealing with the specific analysis should be followed for detailed guidance. These SOPs have also incorporated USACE guidance as described in Section 9.4. Multi-concentration initial calibration (ICAL) using multiple internal standards is performed upon instrument startup and whenever continuing calibration fails the acceptance criteria. A five (or more) point standard curve is prepared using calibration standard solutions that contain all the target compounds, surrogates and internal standards, if any. The concentration of the low standard should be at the method quantitation limit (MQL), and the high standard defines the usable linear range of the detector. After the five or more standards are analyzed, a calibration curve is generated using either the internal standard or the external standard methodology. Ideally, all volatile compounds should exhibit enough linearity for a straight line fit (linear regression), a response factor (RF) or calibration factor (CF) procedure. However, some compounds may exhibit true but consistent non-linearity, which may be handled using a quadratic fit provided sufficient standards (six to seven) are analyzed. The analyst should visually inspect the curves before proceeding with sample analysis. Due to the complexity associated with the volatiles analysis, AML SOP dealing with the specific analysis should be followed for detailed guidance.

An alternative to quantitation from a calibration curve is quantitation from an average response factor (RF) or calibration factor (CF). This is the preferred technique for SW-846 methods (8000 series), 40 CFR 136 methods (600 and 1600 series), and drinking water methods (500 series). For the 8000-series methods, the average RF or CF may be used for sample analysis, if the %RSD for the initial calibration is within the QC limit specified in the EPA method or the applicable AML SOP. The QC limits are different for GC and GC/MS methods and they also vary for different EPA programs. Linear regression should be used when the QC limit for %RSD is exceeded. Non-linear regression may be used if correlation coefficient for linear regression is in excess of the QC limits. Alternatively, the analyst may set up two five-point curves, one to be used low analyte concentrations and the other for high levels of analytes.

Continuing calibration verification (CCV) standards are analyzed at the intervals specified in the methods. The CCV standard concentration is normally at the mid-point of the five-point calibration curve, and to the extent possible at the level specified in the methods. The analyzed value of each standard component should meet QC criteria that are described in the applicable AML SOP and/or EPA method. For this determination, the %difference between the observed RF (or CF) for CCV is compared with the mean RF (or CF) obtained during ICAL. If the CCV standard fails acceptance criteria, another CCV standard may be analyzed. If the second standard also fails, a new initial calibration should be performed after correcting technical problems that might have caused the QC failure.

Several compounds may exhibit erratic behavior. For example, permanent gases such as chloromethane, freons, etc. exhibit wider deviations. The compound, 2-Chloroethyl vinyl ether exhibits erratic chromatographic behavior. For example, the standards vendor, Supelco has added the following footnote to the Purgeable A Standard Mixture: "Due to instability of 2-chloroethyl vinyl ether, we cannot guarantee the concentration of this component." Problems such as this may impact the ability of the laboratory to consistently analyze for this compound within the method requirements or MQL. Similar problems may also arise for common laboratory contaminant compounds such as acetone, methylene chloride, etc. and compounds that are difficult to purge, extract and/or to chromatograph (acids, bases, alcohols and ketones). If calibration or MQL requirements cannot be met, an appropriate qualification flag (e.g. J) should accompany data for these compounds in the report.

9.4.3.2 GC Semivolatiles/Pesticides/PCBs /Herbicides

The SOPs that deal with these analyses have incorporated USACE guidance as described in Section 9.4. Initial calibration (ICAL) is performed upon instrument startup and whenever a CCV standard fails the acceptance criteria. A five (minimum) or six-point standard curve is prepared using all target compounds. The concentration of the low standard should be at the method quantitation limit (MQL), and the high standard defines the usable linear range of the detector. After all the ICAL standards are injected, the computer software generates a calibration curve using either internal standard or external standard methodology. The analyst chooses the best fit type for each compound, either linear or quadratic. The analyst should inspect the curves before proceeding with sample analysis.

The preferred alternative to quantitation from a calibration curve is quantitation from an average response factor (RF) for internal standard methods and average calibration factor (CF) for external standard

methods. These procedures may be used as long as method specific minimum %RSD criteria are met. Most GC methods are dual column methods. These methods also contain procedures for single component parameters (e.g. aldrin, alpha-chlordane, etc.) and multi-component parameters (e.g., PCBs, technical chlordane, etc.) The QC requirements apply to data collected on each column. Due to the complexity associated with each parameter, AML SOP dealing with the specific analysis should be followed for detailed guidance.

Continuing calibration verification (CCV) standards are analyzed at the intervals specified in the methods. If the %D between the RF or CF obtained during CCV and the mean RF or CF obtained during ICAL is within QC limits, the ICAL is acceptable for quantitating sample concentrations. If the CCV standard fails acceptance criteria, another CALV standard may be analyzed. If the second standard also fails, a new initial calibration should be performed after corrective action is performed to rectify the underlying problems.

Calibrations for samples analyzed in accordance with the EPA contract laboratory program (CLP) statement of work (SOW) protocol may differ from those used with other EPA methodologies. For CLP analyses, calibration curves with a minimum of three points are kept on record at the lab. The CLP statements of work (OLM 2.0, OLM 3.0, OLM 4.0, etc.) are followed as written.

9.4.3.3 Total Petroleum Hydrocarbons (Purgeable and Extractable)

A number of State methods exist for determining volatile (OA-1, MADEP VPH, GRO, etc.), extractable (OA-2, MADEP EPH, DRO, FL-PRO, etc.) and total extractable petroleum hydrocarbons in addition to the guidance provided in SW-846 Method 8015. AML has adopted a combination of the 8015 and the most commonly used State method (OA1 and OA2) to streamline operations. Individual diesel range organic (DRO) standards (C-10 to C-28 straight chain hydrocarbons) or gasoline range (GRO) standards at five concentration levels and/or petroleum product standards (e.g. Diesel #2, gasoline, etc.) are used for initial calibration. The %RSD for the initial calibration (ICAL) must be within QC limits for the initial calibration to be valid. In addition, standards of several common commercial petroleum products such as Diesel #2, #4, and #6, Fuel Oil #2, #4 and #6, Kerosene, Jet Fuel #4, lubrication oil, hydraulic fluid, VM&P naphtha, mineral spirits, etc. are run at a single concentration level. The continuing calibration consists of verifying the initial calibration with a mid-level standard. For the ICAL to be acceptable, the percent difference (%D) for the observed CF from CCV from the mean CF for ICAL should be within QC limits. Integration procedures and the calculation of sample results should be in accordance with specifications from appropriate state/federal/client method.

9.4.4 Gas Chromatograph/Mass Spectrometers (GC/MS)

Hardware tuning is performed on each GC/MS prior to calibration as specified in the applicable EPA methods. Ion abundance acceptance criteria for volatile GC/MS tuning with BFB and semivolatile GC/MS tuning with DFTPP are shown in Tables 9.4 and 9.5 respectively. Mass calibration is performed as an integral part of tuning. Tuning is performed at the beginning of each 12-hour period for each GC/MS in accordance with EPA methods. The chromatographic column performance is also verified by analyzing by checking DDT breakdown and peak tailing for difficult compounds such as pentachloropheol.

The SOPs that deal with these analyses have incorporated USACE guidance as described in Section 9.4. Initial calibration (ICAL) based on multiple internal standards is performed at instrument startup and whenever a continuing calibration verification standard (CCV or CCV) standard fails QC acceptance criteria. A five-point standard curve is prepared by analyzing standard solutions containing all the target compounds at varying levels and the surrogates and internal standards at fixed levels. ICAL concentrations are either those defined in CLP protocols, SW-846 Method 8260B, 8270C, etc. or those chosen by the laboratory or client. Response factors are generated for each compound. The acceptance criteria used to assess the calibration are based on the behavior of selected compounds, which are labeled system performance check compounds (SPCC) and calibration check compounds (CCC). These are specified in SW-846 for the 600- and 8000-series methods and in the various CLP SOWs for CLP analyses. For example, the %RSD for each target analyte should be <15% and the RSDs for CCCs must be <30% during initial calibration for both volatiles and semivolatiles. The minimum response factor (RFs) for SPCCs is compound dependent; they should be ≥ 0.05 for semivolatiles and >0.10 (for chloromethane, 1,1-dichloromethane and bromoform) or >0.30 (for chlorobenzene and 1,1,2,2-tetrachloroethane) for volatiles for initial and continuing calibrations.

The mean %RSD for RF in all the target compounds should not exceed QC limits (15%). Linear regression should be used if the QC limits for %RSD is exceeded. Non-linear regression may be used if correlation coefficient for linear regression is in excess of the QC limits. Alternatively, the analyst may set up two five-point curves, one to be used for low analyte concentrations and the other for high levels of analytes.

The percent difference (%D) between the RFs obtained during CALV should be less than QC limits (20%) from the mean RFs obtained during initial calibration. Additional criteria for acceptable CALV include the following: retention times and areas for internal standards for CALV within QC limits based on the values observed during ICAL. If the CALV standard fails acceptance criteria, another CALV standard may be analyzed. If the second standard also fails, the initial calibration must be repeated after appropriate corrective action is performed to resolve observed problems. Problems encountered for permanent gases and other compounds such as 2-chloroethyl vinyl ether, vinyl acetate, etc. which exhibit poor stability or very poor chromatographic behavior may be outside their control limits. In addition, %RSD and %D may be outside the QC limits for common laboratory contaminants (e.g. acetone, methylene chloride, phthalate esters, etc.) and compounds that are difficult to either purge (alcohols, ketones) or to extract (acids, bases, alcohols and ketones). Appropriate flags should accompany data for these compounds in the report.

9.4.5 High Performance Liquid Chromatography (HPLC)

The SOPs that deal with these analyses have incorporated USACE guidance as described in Section 9.4. The calibration procedures for HPLC procedures are similar to those described for GC procedures. Multi concentration initial calibration (ICAL) is performed at instrument startup, following instrument maintenance or change in conditions, and whenever CALV fails acceptance criteria. Response factors (RF) or calibration factors (CF) are generated either manually or by using the chromatography data system.

9.5 Field Instrument Calibration

Calibration of field instrumentation (conductivity meters, pH meters, DO meters, and turbidimeters) is performed in the field prior to use, in accordance with applicable requirements. Calibration data are documented in a bound field notebook or in appropriate project specific binders.

9.6 Calibration Documentation

Calibration records including raw data, response factors, standard concentrations, curves, reduced data, and instrument settings or conditions are stored and archived according to laboratory standard operating procedures. Current chromatograms, curves, and results transcribed onto forms should be kept at the analysts' workstations and periodically archived into a data storage area. Initial and continuing calibrations may be stored by date for ease of location. Standard ID numbers should appear on graphs, plots, chromatograms or curves for traceability purposes. Since QC data and calibration data are shared by multiple AML work orders and/or multiple client projects, a single procedure that works well for the whole laboratory does not exist. The AML SOP on data storage and retrieval procedures should be followed for detailed guidance.

TABLE 9.1
MAJOR LABORATORY INSTRUMENTS

Instrument	Make & Model Number	Quantity	Analysis Performed
Furnace AA with auto sampler	PE-Zeeman 5100, EDL HGA-600 & AS-60	1	TAL Metals (Sb, As, Pb, Se, Tl)
ICP-AES with Autosampler	TJA-61E	1	ICP TAL Metals
ICP-MS with autosampler	PE-Sciex Elan-6100 CETAX Technologies	1	Metals (ultra-low Concentrations)
Mercury Analyzer With autosampler	Spectro Products Leeman Labs	1 1	Mercury
GC/MS	HP-5890/5970	1	GC/MS Volatiles
Purge & Trap Eqpt.	Tekmar 3000	1	Volatiles
Purge & Trap Eqpt.	Tekmar 3010	2	Volatiles
P&T Autosampler	Tekmar Precept II	2	Volatiles
P&T Autosampler	Varian Archon	3	Volatiles
GC/MS	HP-6890 Series II/5973	2	GC/MS Volatiles
GC/MS	HP 6890 series II /5973	2	GC/MS Semivolatiles
LC/MS	HP-5889A	0	LCMS
LC/MS Interface	Analytica	0	LCMS
GC-FID/NPD	HP-5890	1	TPH-Diesel (OA2)
GC-MS/FID	HP-5890/FID	1	TPH-Gasoline (OA1)
GC-FID	HP-5890	1	Methane, Ethane, etc.
GC-ECD/ECD	HP-5890 Series II	1	Pesticides/PCBs/Herb.
GC-ECD/ECD	HP-5890 Series II	1	Pesticides/PCBs/Herb.
GC-Data System	HP-Chemstation	Several	GC Volatiles
Integrator	HP-3396A	2	GC/HPLC Parameters
Integrator	Spectraphysics-4290	1	GC/HPLC Parameters
GC Autosampler	HP-7673	2	GC 3Tests
GC Autosampler	HP-7683	2	GC & GC/MS Tests
GC Autosampler	HP-7673	1	GC & GC/MS Tests
GC Autosampler	HP-7673A	2	GC/MS Tests
Purge & Trap	OI-4460A	1	GC/MS Volatiles
P&T Autosampler	OI-MPM-16	1	GC/MS Volatiles
Purge & Trap	Tekmar-2000	2	GC Volatiles
P&T Autosampler	Tekmar-2016	1	GC Volatiles
HPLC Pump	Waters-590	1	Explosives, PNA, etc.
HPLC UV Detector	Waters-481	1	Explosives, PNA, etc.
HPLC UV Detector	Waters-486	1	Explosives, PNA, etc.
HPLC UV Detector	Waters-440	1	Explosives, PNA, etc.
HPLC UV Detector	Waters-486	1	Explosives, PNA, etc.
HPLC System	HP-1090 & Autosampler	1	Explosives, PNA, NG
HPLC System	HP-1100 & Autosampler	2	Explosives, PNA, NG

TABLE 9.1 (continued)
MAJOR LABORATORY INSTRUMENTS

Instrument	Make & Model Number	Quantity	Analysis Performed
Autosampler	Waters-712 WISP	1	Explosives, PNA, etc.
Gradient Controller	Waters-680	1	Explosives, PNA, etc.
HPLC Fluorescence Detector	LDC-4100	1	PNA
HPLC Pump	Waters-501	1	Explosive, PNA, etc.
HPLC Equipment	Waters-430 conductance Meter	1	Ion chromatography
HPLC Column heater	BIORAD Model	2	HPLC Analyses
UV-VIS Spectrophotometer	Milton Roy Spectronic 20D	2	Wet Chemistry
Ion Chromatograph	Dionex DX-120	2	Anions
Variable Autotransformers`	Powerstat (3 units)	1	General purpose
TOC	Tekmar Dohrmann Astro 2001 system 2	1 1	Total Organic Carbon
Analytical Balance	LECO LB-80	1	Gravimetric
Analytical Balance	Sartorius & Other	3	Gravimetric
pH/Ion Meter	Fisher Accumet 230	1	pH, Ions
Conductivity/TDS	Orion – 124	2	Conductivity, TDS
pH Meter	Corning 320	2	pH
Flashpoint	Koehler K-14600	2	Flashpoint
Mechanical Shaker	Eberbach 6010	1	Sample Preparation
Hot Plates (2l)	Corning PC-353	Several	General
Hot Plate, 6 position	LabLine	Several	General
Hot Plate	Various Unknown	Several	Metals Digestion
Soxtec	1046/1043	1	Organic Extractions
DO Meter	YSI Model 58	1	BOD, COD and DO
Oven	Thelco Model 4	1	General, gravimetric
Oven	Precision GCA	1	General, gravimetric
Muffle Furnace	Fisher 186A	1	General, ash
BOD Incubator	Precision Scientific-815	1	BOD
Ultrasonic Disruptor	Tekmar	3	Sample Preparation
TKN Digestor	SI-AD4020 and Labconco RapidStill II	1	TKN
Sonication Bath	Tekmar	1	Org. Sample Prep.
Water Bath	Tekmar & Other	3	Temperature Control
Water Bath	Neslab CFT-25	2	Temperature Control
Cyanide Distillation	MIDI	1	Cyanide

TABLE 9.1 (continued)
MAJOR LABORATORY INSTRUMENTS

Instrument	Make & Model Number	Quantity	Analysis Performed
Centrifuge	Baxter Medifuge 3632	1	Sample Preparation
Sonicating bath	Branson	2	Explosives Analysis
Laboratory Hoods	Various makes and models	8	Sample Preparation
Laboratory Hood		1	Sample Receiving
Still	Barnstead MP-1	1	General Use
TCLP Tumblers	Analytical Testing	2	TCLP, all parameters
TCLP ZHE	Associated Design, etc.	6	TCLP Volatiles
Autoclave	Peltsen & Crane Model OCM	1	Biological Sample Disposal
Nitrogen Evaporator	Organomation N-Evap Model 112	2	Organic Extractions
Hot Plate	Fisher	Several	Sample Preparation
pH Meter	Fisher Accumet 925	1	pH Measurements
Water Bath (10L)	Fisher	1	General Purpose
Walk-In Refrigerator	Hussman Model KN	1	Sample Storage
Refrigerator	REVCO	1	VOC Sample Storage
Pure water System	USFilter PureLab Plus	1	Type I/II Water
Photocopier	Various	2	Reports/Copying
Copier/Scanner (high-speed)	Various	1	Reports/Copying
High Speed Facsimile	Okidata Okifax 2350 Intellifax 1270	1	Data Reporting Communications
Laser Printer	Canon LBP-860	1	Report Preparation
Laser Printer	HP Laserjet 5	1	Report Preparation
Laser Printer	HP-4050 TN	1	ICP/MS data handling
Laser Printer	HP-4050	1	Report Preparation
Notebook PCs	Various makes and models	3	Data handling
Personal Computers	Various makes and models	Several	Report Preparation
Personal Computers	Various makes and models	>20	Data Processing Various tests
Miscellaneous Comp. Equipment	HP 9122; HP 7957; HP 9144; HP 9000/300	1	Data Processing
Network Server	Dell PowerEdge 4300	2	LIMS Hardware
LIMS Software	Chemware Horizon	1	LIMS Software
Data Storage	Quantum Snap Server	1	Data archive/retrieval

TABLE 9.2
PARTIAL LIST OF STANDARDS - PARAMETER, SOURCE AND STORAGE

Parameter	Source	Stock Standard Type	Storage of Standard
Metals (ICP & AA)	Spex, Solutions Plus	1000 µg/ml solutions	Room Temperature
Metals (ICP & AA)	Ricca, Ultra, etc.	1000 µg/ml solutions	Room Temperature
Metals (ICP & AA)	Solutions Plus, etc.	Mixed Metal Standards	Room Temperature
VOCs (GC/MS)	Ultra Scientific, etc.	IS: 1000 µg/ml mix	Freezer
		SS: 1000 µg/ml mix	Freezer
		MS: 1000 µg/ml mix	Freezer
		BFB: 2000 µg/ml soln.	Freezer
		CAL: 100 µg/ml mix.	Freezer
SVOCs (GC/MS)	Ultra Scientific, etc.	IS: 4000 µg/ml mix	Refrigerator
		Acids SS: 1500 µg/ml mix	Refrigerator
		BNA SS: 1000 µg/ml mix	Refrigerator
		MS: 1500-2000 µg/ml mix	Refrigerator
		DFTPP: µg/ml mix	Refrigerator
		CAL: 1000 µg/ml mix	Refrigerator
Pesticides/PCBs (GC)	Ultra Scientific, etc.	PCBs: 100 µg/ml Aroclors	Refrigerator
		Pest: 250 µg/ml cal mix	Refrigerator
		MS: 50-100 µg/ml mix	Refrigerator
Other SVOCs & other Pesticides (GC or GC/MS)	Chem Service Supelco Ultra	Neat Standards Individual Solutions Method solution mix	Refrigerator
	Ultra Scientific	Method mix standards	Refrigerator
	NSI	Ind. solns. 1000 µg/ml	Refrigerator
	Supelco	Method mix standards	Refrigerator
Explosives	Ultra Scientific AccuStandard, etc.	CAL: 1000 µg/ml mix	Refrigerator
PH/Conductivity	Fisher, Baxter	Cal. buffer solution Cal. standard solution	Room Temperature
Anions & Cations	Fisher, Baxter, Taylor, Others	Neat materials	Room Temperature
Other Parameters	Sigma, Aldrich, Fisher, Others	Neat materials, solutions	inorganics - room temp. organics - refrigerator
Refrigerators (5)	Various makes, Models and sizes	10-1-94	Sample and Standard Storage

TABLE 9.2 (continued)
PARTIAL LIST OF STANDARDS - PARAMETER, SOURCE AND STORAGE

Parameter	**Lab. Stock and Working Standard**	**Storage of Lab Stock and Working Standard**	**Frequency of Prep., LabStock/working std.**
Metals (ICP & AA)	From Stock	Room temperature	Quarterly/as needed
VOCs (GC/MS)	From Stock	Freezer / refrigerator	Quarterly/weekly
SVOCs (GC/MS)	From Stock	Refrigerator	Semiannually/as needed
Pesticides/PCBs (GC)	From Stock	Refrigerator	Semiannually/as needed
Other SVOCs Pesticides	From Stock	Refrigerator	Semiannual/as needed
Explosives	From Stock	Refrigerator	Quarterly/weekly
pH/Conductivity	Used as is	Room Temperature	Annually
Anions & Cations	From Stock	Room Temperature	As needed
Other Parameters	From Stock	Inorganics: Room temp. Organics: Refrigerator	As needed

TABLE 9.3
PREPARATION OF STANDARD SOLUTIONS

Type of Stock	**Method of standard solution preparation**
Neat Material	Weigh out or measure out and dilute to known volume in a suitable solvent in class A volumetrics to prepare concentrated stock solutions.
Concentrated stock solutions	Dilute known volumes of the stock solutions with a suitable solvent to a known final volume to prepare intermediate standards.
Intermediate standards	Dilute known volumes of the intermediate or stock solutions with a suitable solvent to a known final volume to prepare working standards.
Working standards	Used as is and stored for shorter time periods; organic standards including volatiles are stored in the refrigerator.

Table 9.4

Volatile organic analysis tuning criteria

Mass	Bromofluorobenzene (BFB) ion abundance criteria
50	15.0 - 40.0% of mass 95
75	30.0 - 60.0% of mass 95
95	Base peak, 100% relative abundance
96	5.0 - 9.0% of mass 95
173	Less than 2.0% of mass 174
174	Greater than 50.0% of mass 95
175	5.0 - 9.0% of mass 174
176	Greater than 95.0%, but less than 101.0% of mass 174
177	5.0 - 9.0% of mass 176

Table 9.5

Semivolatile organic analysis tuning criteria

Mass	Decafluorotriphenylphosphate (DFTPP) ion abundance criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

10.0 PREVENTIVE MAINTENANCE

Routine and non-routine maintenance are performed on equipment in order to gain maximum performance and minimal downtime. Most major equipment utilized by the laboratory is covered by a service contract with a local vendor to minimize response time. An inventory of spare parts is also maintained for each instrument in order to minimize down time. Procedures have been established for subcontracting analytical work in the event of an unscheduled instrument down time.

10.1 Maintenance Schedule

Regular inspection, cleaning, servicing and maintenance of analytical equipment are performed according to the manufacturer's recommendations. The preventive maintenance schedule varies with the type of instrument and is shown in Table 10.1. Preventive maintenance is performed by the analysts responsible for the particular instrument(s).

10.2 Documentation

Routine and non-routine equipment maintenance performed in-house or by a service representative is documented in a maintenance logbook specific to the piece of equipment being maintained. The documented information include: type of maintenance, reason for maintenance, type of malfunction, steps taken to correct the malfunction, date, and name of person performing the maintenance.

10.3 Monitoring

Laboratory ovens, refrigerators, freezers, incubator, balances and water systems are monitored on a regular basis as shown in Table 10.2. For electronic balances, the monitoring program includes a daily check of the calibration. Table 10.3 contains example criteria used for the monitoring program. Detailed criteria are included in the AML standard operating procedure (SOP) on water quality, temperature measurements and gravimetric determinations. The monitoring activities are recorded in the appropriate maintenance logbook.

10.4 Contingency Plans

In the event of instrument failure, portions of the sample load may be diverted to redundant instrumentation within the facility. When permissible, an alternate analytical technique may be used as long as it is acceptable to the client and in accordance with the project plans. Subcontracting is the last option used by the AML. Samples or extracts shipped to another facility will be accompanied by proper chain-of-custody procedures. Subcontract laboratories must meet the quality assurance criteria for the project and must have all required certifications. Samples will only be subcontracted if permitted by contract and with permission from the client.

Table 10.1

PREVENTIVE MAINTENANCE SCHEDULE

Instrument Type	Type of Maintenance	Freq-uency	Perform-ed By
GC	Check gas tanks	daily	(in house)
	Check oxygen/HC/Moisture traps	4/year	(in house)
	Change septum	every day	(in house)
	Replace injector liner	as needed	(in house)
	Clip front end off column	as needed	(in house)
	PID Detector		
	- clean lamp window	as needed	(in house)
	NPD Detector		
	- clean jet tip and collector	as needed	(in house)
	FID Detector		
	- clean jet tip and collector	as needed	(in house)
	Electron capture detector		
	- raise operating temperature to 350°C	as needed	(in house)
	ELCD/Hall Detector		
	- change resin beads	as needed	(in house)
	- monitor and change solvent	as needed	(in house)
GC/MS	*Mass Spectrometer*		
	Change pump oil	4/year	(in-house)
	Clean ion source	as needed	(in-house)
	Clean quadrupole rods	as needed	(in-house)
	Oil turbo pumps	4/year	(serv. eng.)
	verify spectra will meet their specifications	2/year	(serv. eng.)
HPLC	*Pumps:*		
	- change seals	monthly	(in house)
	- change seal retainer PTFE O-ring	3/year	(in house)
	-change/clean check valves	as needed	(in house)
	Detector:		
	- clean cell	monthly	(in house)
	Columns and Lines:		
	- clean inlet solvent filters	monthly	(in house)
	- change solvent tubing	as needed	(in house)
	- replumbing lines	as needed	(in house)
ICP-AES & ICP-MS	Change vacuum pump oil	4/year	(in house)
	windows are cleaned	4/year	(in house)
	Torch cleaned	4/year	(in house)
	Channel alignment checked	as needed	(in house)
	Linear Ranges determined	4/year	(in house)
	Inter-element correction factors determined and applied	4/year	(in house)
	Instrument Detection Limits Determined	annual	(in house)
	Computer System areas cleaned	4/year	(in house)

Table 10.1 (contd.)

PREVENTIVE MAINTENANCE SCHEDULE

Instrument Type	Type of Maintenance	Freq-uency	Perform-ed By
GFAA & CVAA	Optics dusted	2/year	(serv. eng)
	System performance check	2/year	(serv. eng)
	Computer system areas cleaned	2/year	(serv. eng)
	System cleaned and aligned	2/year	(serv. eng)
Autoana-lyzer	Pump tube change	monthly	(in house)
	Inspect and replace any worn parts	monthly	(in house)
	Back-up computer system	monthly	(in house)
	Wipe entire system down with DI water	monthly	(in house)
	replace air filter	2/year	(in house)
Balances	System cleaned	annual	(serv. eng.)
	Checked with independent NBS class "S" weights for linearity and adjusted as necessary	annual	(serv. eng)
	Calibration checks	daily	(in house)
pH Meter	Inspect probe membrane	daily	(in house)
	Check filling solution level	daily	(in house)
Ion Selective electrodes	Inspect & polish electrodes	monthly	(in house)
Cond. Meter	Inspect cell & replatinize if necessary	4/year	(in house)
Ion Chro-matograph	Inspect pump pistons	annual	(in house)
	Check AS3 columns	daily	(in house)
	Check AS3 guard columns	daily	(in house)
DO Meter	Check probe membrane	daily	(in house)
VIS Spec.	Verify wavelength	annual	(serv. eng.)
Ovens	Check temperature	daily	(in house)
Block Dig.	Check temperature	4/year	(in house)
BOD incub.	Check temperature	2/daily	(in house)
waterbaths	Check temperature	daily	(in house)
TCLP equip.	Check rotation rate	4/year	(in house)
TOC Ana-lyzer	Check pump tubes	weekly	(in house)
	Check and adjust flow rates	monthly	(in house)
	Check and clean detector windows	annual	(in house)

Table 10.1 (continued)

PREVENTIVE MAINTENANCE SCHEDULE

Instrument Type	Type of Maintenance	Freq-uency	Perform-ed By
Pipettes, Mechanical	Volume Calibration	see SOP	(in house)
Thermo-, Meters	Calibration	see SOP	(in house)
Reference Standards	Inspect for degradation	see SOPs	(in house)

Table 10.2

EQUIPMENT MONITORING

Equipment Type	**Frequency**	**Procedure**	**Acceptance Criteria**
Analytical Balances	Daily	Check with class S weights (ASTM D5522 Standard)	±0.1 %
Top Loading Balances	Daily	Check with 1 class S weight (ASTM D5522 Standard)	±1%
Class S Weights	Annual	Outside Vendor	ASTM-D5522
Oven (%solids)	Daily	Check and record temperature	104° ± 2° C
Refrigerators	Daily	Check and record temperature	4° ± 2° C
Freezers	Daily	Check and record temperature	-13° ± 2° C
BOD Incubator	Daily	Check and record temperature	20° ± 2° C
Incubator	Daily	Check and record temperature	45° ± 2° C
NIST Thermometer	Annual	Check at 0°C and 100°C	± 0.5°C
DI water system	Daily	Check conductance and record	≥ 1- Mohm-cm at 25°C
Type I Water	Daily	Check conductance and record	≥ 18- Mohm-cm at 25°C

Table 10.3

Balance Calibration Log

Date: _____ Weight Set ID: S01 Analyst Initials: _____

Balance: OHAUS Analytical Plus - Serial Number: 1118133828				
True Weight TW (g)	**Observed Weight OW (g)**	**Error (g) |OW-TW|**	**Tolerance Limits (g)**	**Corrective Action**
100.0000			± 0.100	
5.0000			± 0.005	
0.2000			± 0.0002	

Balance: LECO LB-80 - Serial Number: 363 (Use is restricted to ± 0.001-g accuracy)				
True Weight TW (g)	**Observed Weight OW (g)**	**Error (g) |OW-TW|**	**Tolerance Limits (g)**	**Corrective Action**
100.0000			± 0.100	
5.0000			± 0.005	
0.2000			± 0.001	Restricted Use

Balance: Sartorius Type 1601-MP8-1 - Serial Number: 3310099				
True Weight TW (g)	**Observed Weight OW (g)**	**Error (g) |OW-TW|**	**Tolerance Limits (g)**	**Corrective Action**
100.0000			± 0.100	
5.0000			± 0.005	
0.2000			± 0.0002	

Acculab LA-200 - Serial Number: 80305394				
True Weight TW (g)	**Observed Weight OW (g)**	**Error (g) |OW-TW|**	**Tolerance Limits (g)**	**Corrective Action**
100.0000			± 0.100	
5.0000			± 0.005	
0.2000			± 0.0002	

Other Balances (describe):				
True Weight TW (g)	**Observed Weight OW (g)**	**Error (g) |OW-TW|**	**Tolerance Limits (g)**	**Corrective Action**
100.0000			± 0.100	
5.0000			± 0.005	
0.2000			± 0.0002	

11.0 QUALITY CONTROL CHECKS

A successful quality assurance program requires routine assessment of quality. Data quality is judged in terms of its precision, accuracy, representativeness, completeness and comparability. AML uses quality control samples such as blanks, spikes, laboratory control samples, known standards, check samples and standard reference materials as internal quality control checks. Routine application of this program requires initial development, periodic updates and successful implementation of the overall quality assurance program.

11.1 Development of Quality Control data

This section describes the process of how quality control data is generated during the routine analysis of samples.

For most tests, an initial calibration curve consisting of at least three standards and a reagent blank is prepared for each parameter. If the standard curve is known to be stable, the curve can be verified daily by the analysis of a midpoint standard. The percent difference (%D) between the continuing calibration and the initial calibration is measured to validate the initial curve. See applicable AML SOPs for method-specific calibration procedures.

A method blank (MB) is prepared and analyzed with each set of samples. Field blanks (when provided by clients) are analyzed to determine possible sample contamination during collection and shipment to the laboratory. Field blanks such as trip blanks are applicable to volatile organics analysis (VOA) where volatile contaminants can be introduced from ambient air on site, during shipment, and in the laboratory.

A laboratory control sample (LCS) or a quality control check standard (QC) consisting of deionized water or clean laboratory sand spiked with the parameter of interest is analyzed for each matrix and each batch of analysis. The recoveries of individual analytes are used to set up control charts. The LCS is analyzed in duplicate (LCSD) if batch-specific precision data is needed and/or sample duplicates or matrix spike (MS) duplicates (MSD) are unavailable.

One sample is to be analyzed in duplicate for each matrix and each batch of analysis. The minimum frequency for duplicates will be one for each twenty samples. The percent difference between duplicate measurements is used to assess precision. Matrix spike duplicates are used instead of sample duplicates to maximize data availability for target analytes. LCS duplicates are used as substitutes when environmental samples are unavailable to perform MS and MSD determinations.

One sample is to be spiked preferably at a level equal to or greater than the original concentration of the sample before dilution and analysis for each matrix and each batch of analysis. A matrix spike duplicate will also be prepared for each sample selected for spiking. The minimum frequency for matrix spikes will be one for each ten samples.

The recoveries of individual analytes and the percent difference between duplicate matrix spike measurements are used to set up control charts. Matrix spike recoveries may or may not be

meaningful if information on expected sample results were unavailable at the time of matrix spike analysis. Matrix spike analyses may not be performed if sufficient sample volumes are not collected.

The QA Director may provide blind samples from time to time to evaluate analyst performance. Blind samples are routinely used for the analysis of metals, water quality parameters, and organic analyses, which do not require separate extractions. Standard Reference Materials (SRMs) are introduced periodically into the testing scheme by the QA Manager to evaluate the testing procedure and the analyst's performance.

Samples designated for most organic analyses are spiked with the appropriate surrogate standards prior to extraction and analysis for volatile and semivolatile organic compounds, pesticides, PCBs, etc. Internal standards are added to all sample extracts prior to GC/MS analysis.

The acceptance of analytical results and the control of analytical systems are based on the evaluation of quality control data. The quality control program that is established at AML adheres to the requirements of the US Environmental Protection Agency. The "Handbook for Analytical Quality Control in Water and Wastewater Laboratories" (EPA 600/4-79-019) is used as one of the reference sources for the quality control program at AML.

Accuracy is the measure of the closeness of an observed value to the true value, which could be a reference value, theoretical value or the true population mean. Accuracy has two components: precision, which is a measure of random error and bias, which is a measure of systematic error. Initial precision and bias data are generated through repetitive analyses of a series of artificially contaminated samples. This series encompasses the anticipated concentration range of routine samples and the linear concentration range of the analytical procedure. The control limits and mean recoveries determined by this process and internal or external administrative guidelines form the basis to evaluate the validity of each specific set of analytical results. The procedures used by AML for development of these criteria, evaluation of analytical data, and making decisions of appropriate action are presented in the following sections.

11.2 Precision

To determine the initial precision of an analytical procedure, a program of analyses of replicate samples of an appropriate concentration range is conducted. In order to establish the upper and lower control limits (UCL and LCL) for a particular analysis within a specified concentration range, a minimum of 20 sets of replicate analysis is performed. The results of the replicate analyses are used to calculate the relative percent difference (RPD), which is defined as 100 times the difference (range) of each replicate set, divided by the average value (mean) of the replicate set. For replicate results D_1 and D_2, the RPD is calculated by.

$$RPD = \frac{|D_1 - D_2| * 100}{(D_1 + D_2) * (0.5)} \qquad \text{(Equation 1)}$$

When the RPD is obtained for at least twenty replicate pairs, the mean RPD and the standard deviation are calculated using equations 7 to 9 in Section 12 of this manual. Quality control (QC)

limits for RPD may be set as described in Section 11.3. Regulatory agencies and laboratory clients may set targets for RPD based on method quality objectives and/or data quality objectives for specific programs or projects. In most cases, QC limits used by the laboratory are derived from these external requirements.

When RPDs are outside QC limits, the analysis may be interrupted. The most common problem is sample heterogeneity for soil samples and incorrect spike amounts for spiked samples relative to their original amount. The laboratory may not always have control over these issues. If the problem is resolvable by the laboratory, and the problem is resolved, analysis can be resumed.

11.3 Bias

In addition to the generation of initial precision data for each method, a system for monitoring bias in laboratory measurements is also maintained to determine the accuracy of data. The data dealing with bias is collected on a routine basis through the interjection of laboratory control samples (LCS and LCSD), spiked samples (MS, MSD, and surrogates) into the analytical stream. This data is compared to initial percent recovery data that has been compiled from the statistical evaluation of recovery results. The initial recovery data is collected from repetitive analysis of a range of spiked samples. The percent recovery (%R) is calculated as follows:

$$\%R = \frac{A_o * 100}{A_t} \qquad \text{(Equation 2)}$$

where

A_o = is the observed amount and
A_t = is the true amount for LCS and QC check samples

Percent recovery (%R) for matrix spike samples is calculated as follows:

$$\%R = \frac{(A_{as} - A_{bs}) * 100}{A_s} \qquad \text{(Equation 3)}$$

where

A_{as} = is the observed amount in the sample after spike
A_{bs} = is the observed amount in the sample before spike
A_s = is the amount spiked

Percent recovery (%R) of surrogate compounds is calculated as follows:

$$\%R = \frac{A_o * 100}{A_s} \qquad \text{(Equation 4)}$$

When the %R is obtained for at least twenty spike recovery analyses, the mean (X_m) and the standard deviation (S) are calculated as described in Section 12 of this plan. Warning (2-sigma) and control (3-sigma) limits, which approximate the 95% and 99% confidence interval respectively, are calculated as follows:

In-house Upper Control Limit (UCL)	=	$X_m + 3*S$	(Equation 5)
In-house Upper Warning Limit (UWL)	=	$X_m + 2*S$	(Equation 6)
In-house Lower Warning Limit (LWL)	=	$X_m - 2*S$	(Equation 7)
In-house Lower Control Limit (LCL)	=	$X_m - 3*S$	(Equation 8)

Additionally, "range" control charts may be used to evaluate interbatch QC data. Range control charts set the 95% and 99% confidence intervals at 2.456-sigma and 3.286-sigma respectively.

A visual control chart is established by plotting the %R for each target analyte on a graph or on a spreadsheet. The average of the %R determinations for the original data set is established as the midpoint on the Y-axis of the graph. The upper control limit and upper warning limits are plotted as solid and dotted horizontal lines respectively across the graph at their respective points on the Y-axis above the midpoint. The lower control and warning limits are plotted as solid and dotted horizontal lines respectively across the graph at their respective points on the Y-axis below the midpoint. An electronic spreadsheet (e.g. Excel) may be used instead of manual graphing procedures.

The calculated %R of each target analyte from routine sample analysis is plotted on the graph to determine whether the %R is within the warning and control limits of the chart. This procedure is performed independently for LCS, MS and surrogate recoveries. If the observed %R plots between the warning and control limits, the Group Leader, Laboratory Director, or Quality Assurance Officer is notified for a decision as to how to proceed. If the observed %R plots outside the control limits, the analysis may be stopped until the source of the problem has been determined and corrective action has been taken.

Since most analytical methods have multiple target analytes (e.g. over 60 for VOCs and SVOCs), it may not be statistically possible to have all the target analytes within QC limits. Minor but constant changes in extraction reagents, extraction conditions, chromatographic column performance, changes in common laboratory contaminant levels, instrument detector performance, multitude of instruments, multitude of personnel, etc. may affect the strict applicability of static charts to make absolute decisions on a daily basis. For example, a single test (e.g. VOCs) may require two control charts for water matrices (5-ml and 25-ml purge) and two additional charts for soil matrices (low-level and medium-level) for each instrument. A medium sized commercial laboratory may have upwards of five GC/MS units performing VOCs analysis, which may require 20 control charts. The observed recoveries for surrogates and matrix spikes are heavily dependent on matrix, which may or may not parallel the control matrix. The preponderance of control charts tend to make project planning very difficult since most geologists and engineers involved in it either do not understand or loathe the complexity. Dynamic control charts may be harder to implement due to high operational cost and business constraints. Dynamic control charts also cause additional confusion for project planners.

Regulatory agencies and laboratory clients may set targets (control limits or QC limits) for %R based on method quality objectives and/or data quality objectives for specific programs or projects. In most cases, default QC limits used by the laboratory are derived from these external requirements. The QA Director has the authority to institute administrative control limits that are based on in-house statistical limits, simpler-to-use administrative limits based on client specifications (e.g. USACE MQOs contained in Appendix I of EM 200-1-3 or Department of Defense Quality System Manual, Revision 2 Final). The QA Director may also institute a combination of the two methods for controlling data quality, when applicable.

11.4 Control Charts

Control charts are developed to predict trends (positive or negative) in the analytical processes and to determine when an analysis is out of control. Examples of situations, which may show up in control charts, are:

- Shift in the mean - this may be caused by incorrectly prepared standards or reagents, contamination of sample, problems in instrument calibration, or analyst error.
- Upward or downward trend of the Mean - this may be caused by deterioration of standards or reagents, increased concentration of the standard due to evaporation of solvent or a steady shift in calibration in one direction, or changes in chromatographic behavior.
- Increase in variability - this may be caused by poor technique of analyst or deviation from procedure.

Control charts are useful in studying processes and in implementing appropriate corrective action, when the process appears to be "out-of-control". For example, a process is said to be “out-of-control” if one or more points fall outside of the control limits. One should check for potential problems if

(1) two out of three successive points between 2σ and 3σ zone on the same side of the mean,
(2) four out of five successive points between 1σ and 2σ zone on the same side of the mean,
(3) nine successive points on one side of the mean,
(4) six consecutive increasing or decreasing points,
(5) fourteen points in a row alternating up and down,
(6) fifteen points in a row within $\pm 1\sigma$ zone, etc.

Although the theoretical principles outlined above are sound, cost and schedule constraints on laboratory clients for environmental projects may not allow the laboratory to allocate sufficient time and resources to implement a detailed control charting program in the laboratory. Also, there is no industry consensus on either what constitutes an "out-of-control" situation requiring corrective action and what constitutes an "appropriate" corrective action. Client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints.

11.5 Method Validation

Any new method or any method being performed by an analyst for the first time should be approved by a validation process. This process is also required whenever extremely difficult or unusual sample matrices are encountered or extremely low detection Units are required. Any method modification also requires method validation before results of the analysis will be released.

Method validation involves analysis of a sample for determination of accuracy, precision, method detection limit and an adequate percent recovery. Data from this validation is kept for each analysis performed and maintained in a separate file. This file contains the method reference, detailed instructions for analysis including any special notes or precautions. Quality control data from the initial method validation, which provides evidence of analyte identification, adequate recovery, accuracy and precision data, and a control chart which tracks continued method performance are kept on file.

Method detection limits (MDL) will be determined during method validation and it will be updated on an annual basis. Low level spikes, initial calibrations, and calibration verification procedures performed on a day-to-day basis will serve as continuing verification of the method detection limits.

11.6 Detection, quantitation and reporting limits

11.6.1 Instrument Detection Limits (IDL)

Instrument detection limits (IDL) are defined as the minimum concentration of a substance that can be measured and reported in reagent water with 99% confidence that the substance concentration is greater than zero. IDLs can be estimated as 2.5 to 5 times the signal to noise (S/N) ratio for the instrument. IDLs are determined from actual analysis of spiked water samples and they are normally used for reporting metals detection limits for some programs. IDLs are measured from replicate analysis (3 to 10) of blank and/or standard solutions close to the estimated instrument detection limit. Calculate IDL as described for method detection limits (MDL) in Section 11.6.2. IDLs will vary depending on the amount of standard injected, instrument condition, instrument tune and the mode in which it is operated. The AML standard operating procedure (SOP) that governs IDL, MDL, RL and PQL should be used for additional details.

11.6.2 Method Detection Limits (MDL)

Method detection limits (MDL) are defined as the minimum concentration of a substance that can be measured and reported in a specific matrix and by a specific method with 99% confidence that the substance concentration is greater than zero. MDLs are determined from actual analysis of samples in the given matrix and the given method. This procedure as described in 40 CFR Part 136 - Appendix B includes analyzing seven prepared matrix spikes at levels three to five times the estimated method detection limit. The standard deviation, S (see Chapter 12 of this document) of the seven replicate measurements is calculated and the MDL is calculated by multiplying this standard deviation by 3.14 (the Student's t value for 99% confidence interval for 7 determinations).

$$MDL = 3.14 * S \qquad \text{(Equation 9)}$$

The AML standard operating procedure (SOP) that governs IDL, MDL, RL and PQL should be used for additional details.

11.6.3 Method Quantitation Limits (MQL)

Practical quantitation limits (PQL), estimated quantitation limits (EQL), method quantitation limits (MQL) or limits of quantitation (LOQ) are terms that are used to define the lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. These terms have been confusing since some laboratories also use the term reporting limit (RL) synonymously with quantitation limits. Some of the laboratories that use RL synonymously with QL do not report any sample result below the RL. Some laboratories report data below their RL as an estimated quantity.

Quantitation limit is the minimum concentration of a substance that can be quantitated accurately in a specific matrix and by a specific method. The actual definitions for LOQ, EQL, MQL, and PQL may vary widely in the literature. The MQL is defined in some EPA documents (SW-846 Method 8000C, Section 11.4.1.2, Revision 3, March 2003) as the lowest concentration calibration standard that is analyzed during an initial calibration. The U.S. Army corps of engineers (USACE) establishes MQL at a factor of five to ten times the MDL for the majority of target analytes, but no lower than three times the MDL for any target analyte. EQLs, according to EPA may be nominally chosen to simplify data reporting and for many analytes it is chosen as the lowest non-zero standard in the calibration curve.

The AML laboratory uses the concentration of the lowest non-zero standard in the calibration curve as the MQL in accordance with SW-846 Method 8000C. For certain analytes that are difficult to analyze (e.g. ketones or common laboratory contaminants for VOCs analysis), the concentration of the next higher non-zero standard on the calibration curve is used as MQL. For metals analysis by ICP-AES or ICP-MS, MQL is defined as the concentration level that results in an accuracy of ±20%. All target analyte values detected and reported below MQL are flagged (J code) as estimated quantities. The AML standard operating procedure (SOP) that governs IDL, MDL, LLR (see next section) and MQL should be used for additional details. The parameter-specific SOPs should also be used as guidance documents.

11.6.4 Lowest Level of Reporting (LLR)

The lowest level of reporting (LLR) is related to MDLs and MDL check sample analysis. For multi target analyte organic analytical methods such as SVOCs analysis by SW-846 method 8270C, the laboratory may use cut-off levels that may be greater than or equal to the individual target analyte MDLs in instrument quantitation reports. A single cut-off level may be used for all target analytes. This cut-off level is a uniform low level for reporting (LLR) sample results.

When sample concentration for an analyte is greater than LLR, the compound is coded as present only if it is also greater than the associated adjusted MDL. Otherwise, it is coded as not detected (U code). When the sample concentration is less than LLR, it is coded as not detected (U code), even if the observed level is greater than the adjusted MDL. The uniform low level for reporting (LLR) is included in report headers along with information such as dilution factors, sample weight, etc. LLRs are normally corrected for percent solids, sample size and dilution.

LLRs may be used as optional reporting tools by the laboratory. As a general rule, LLRs are normally determined by rounding up the highest MDL for the target analytes to a round number. While the LLRs may equal MDLs when low detection limits are required, they must be less than or equal to MQLs. Professional judgment should be used to determine LLRs. The use of LLR is strictly an optional procedure that may be used in selected projects to simplify laboratory analysis and reporting.

11.6.5 MDL Check Sample Analysis

MDL check samples are prepared by spiking an interference free matrix with all target analytes at approximately two times the MDLs. Actual level spiked at AML should equal the low level for reporting (LLR). The MDL check sample should be taken through the same process used for sample analysis and those initially used to establish MDL values. If any of the target analytes are not detected, then the LLRs should be modified and repeated for the affected analyte(s), until the analytes are detectable in the MDL check sample. MDL check samples are analyzed on a quarterly basis, in lieu of the annual MDL study. When multiple instruments or confirmation columns are used for the same method, separate MDL studies may be replaced by the analysis of a single MDL check sample on all instruments/columns. The MDL check sample should be analyzed after major instrument maintenance, or changes in instrumentation or instrumental conditions to verify the current sensitivity of the method.

11.6.6 Reporting Limits

Laboratory reporting limits (RL) are defined as the minimum concentrations below which the presence of a substance is not reported. Reporting limits are generally used by AML to meet client requirements for simpler projects (level II reports) that do not require detailed reports (Level III or IV). The substance may or may not be present in the sample at concentrations up to the reporting level. Reporting levels must be equal to or greater than MDLs. The AML standard operating procedure (SOP) that governs IDL, MDL, RL, LLR, and PQL should be used for additional details.

11.7 Client Specified Method Quality Objectives

Client specified method quality objectives (MQOs) are quality control criteria specified by clients for their projects. For example, the United States Army Corps of Engineers (USACE) has MQOs that are incorporated into Appendix I of EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans (1 Februrary 2001). In addition to MQOs specified by USACE center of excellence (CX), USACE districts may have more detailed MQOs as part of their data validation guidelines (Kansas City District) or other documents (e.g. quality assurance plans, contract documents (Louisville District), etc.). The Air Force Center for Environmental Excellence (AFCEE) has a detailed quality assurance project plan (Revision 3.1, August 2001) that contains MQOs for a number of EPA SW-846 methods. The MQOs from a number of clients are included in the AML standard operating procedure (SOP) appendices. The MQOs may be treated as project specific requirements that may be in lieu of or supplemental to the laboratory (AML) and/or EPA defined method requirements.

The Department of Defense (DoD) has developed a Quality System Manual (QSM) for environmental laboratories (Final Version 2, June 2002) that specifies QC limits for most common

tests performed in support of army, navy and air force projects. The QC limits included in the DoD QSM are realistic and they are attainable by most environmental laboratories since they are actually based on actual analytical data. The limits are also analyte-specific with allowance for difficult analytes in multi-analyte test methods. AML currently uses the DoD QSM QC limits with minor modifications as default limits for common test parameters.

12.0 DATA REDUCTION, REVIEW AND REPORTING

Achieving the objectives of AML requires the implementation of a system for data reduction, data review, data verification and monitoring for accuracy, completeness, and precision in analysis. Quality is assessed from data on blanks, calibration curves, duplicates, spikes, internal standards or surrogates. Additionally, system audits, laboratory control samples or known standards, and blind standards are used to validate the correctness of sample results. See applicable generic and/or method specific AML standard operating procedure (SOP) for more details on this topic. The information gathered from review is used to determine the laboratory's ability to perform accurate analysis for a given test. Through the use of checklist, charts, performance evaluation and chain of custody procedures, the entire system of data production is verified.

12.1 Data Reduction

Data obtained by the following method/instrument are directly reportable: Volatile GC/MS, volatile GC, semivolatile GC/MS, ICP metals, AA metals, pH, turbidity, automated colorimetry, etc. Factors that affect the final results such as sample weight/volume, final extract or digestate volume, percent solids and dilution factor are input into the instrument computer and correct results are calculated automatically. Manual calculations may be made if any of these correction factors were unavailable at the time of instrumental analysis.

Data requiring reduction prior to reporting include semivolatile GC, PCBs, BOD, COD, conductivity, manual UV/VIS/IR, residue, titrimetric methods, etc. Spreadsheets are used to input relevant information such as calibration curves, sample weight or volume, dilution factors, final volume of the extracts, percent moisture etc. and correct results are calculated in batches.

The following equations are used to calculate results:

$$CF = \frac{A_s}{C_s} \qquad \text{(Equation 1)}$$

where

CF = Calibration Factor
A_s = Peak area of the standard
C_s = Concentration (e.g. µg/ml) of the standard solution

Calibration Factors (CF) are used in volatile and/or semivolatile GC/HPLC analyses of target compounds by external standard calibration methods. Sample concentrations are determined using the following equations for water and soil samples with the assumption that injection volumes are identical for sample extracts and calibration solutions:

$$\text{Amount } (\mu g/L) = \frac{A * V_e * DF}{CF * V_s} \qquad \text{(Equation 2)}$$

where

A = Peak area for the sample extract
V_e = Final volume of the extract (ml) before dilution
V_s = Volume of water sample (L) extracted
DF = Dilution Factor, if any for instrumental analysis

$$\text{Amount } (\mu g/Kg) = \frac{A * V_e * DF * 100}{CF * W_s * PS} \qquad \text{(Equation 3)}$$

where

W_s = Weight of sample (Kg) extracted
PS = Percent solids expressed in percent

$$RF = \frac{A_s * C_{is}}{C_s * A_{is}} \qquad \text{(Equation 4)}$$

where

RF = Response Factor
A_s = Peak area of the standard
C_s = Concentration (µg/ml) of the standard solution
A_{is} = Peak area of the internal standard
C_{is} = Concentration (µg/ml) of the internal standard solution

Response Factors (RF) are used in volatile and semivolatile GC/MS analyses and in some GC/HPLC analyses of target compounds by internal standard calibration methods. Sample concentrations are determined using the following equations for water and soil samples:

$$\text{Amount } (\mu g/L) = \frac{A * C_{is} * V_e}{A_{is} * RF * V_s} \qquad \text{(Equation 5)}$$

where

A = Peak area for the analyte in the sample extract
A_{is} = Peak area of the internal standard in the sample extract
C_{is} = Concentration (µg/ml) of the internal standard in the sample extract
V_e = Final volume of the extract (ml) including any dilution

V_s = Volume of water sample (L) extracted

Equation 6 is used to calculate sample amounts in soil and related matrices.

$$\text{Amount } (\mu g/Kg) = \frac{A * C_{is} * V_e * 100}{A_{is} * RF * W_s * PS} \qquad \text{(Equation 6)}$$

Mean response factors (RF_m) or mean calibration factors (CF_m) are generally calculated during initial calibration (ICAL) from multiple determinations of these quantities as follows:

$$X_m = \sum_{i=1}^{n} X_i \qquad \text{(Equation 7)}$$

where

X= is the mean value of n determinations for parameters such as CF_m and RF_m
Xi = is the individual values for each of n determinations.

Variance (S^2) and standard deviation (S) are calculated using the following equations:

$$S^2 = \{1/(n-1)\} \sum_{i=1}^{n} (X_m - X_i)^2 \qquad \text{(Equation 8)}$$

$$S = \sqrt{S^2} \qquad \text{(Equation 9)}$$

The coefficient of variation (CV) or percent relative standard deviation (RSD) is calculated as follows:

$$CV = RSD = (100 * S)/X_m \qquad \text{(Equation 10)}$$

The percent difference (%D) between a mean value (X_m) and a single value (X_i) such as those obtained during continuing calibration verification (CCV) is calculated as follows:

$$\%D = 100 * \{(X_m - X_i)/ X_m\} \qquad \text{(Equation 11)}$$

Calibration curves for analytes that do not exhibit linearity through zero are constructed through linear or non-linear regression methods. The most common procedure used is a straight-line (linear regression) plot, which uses the following equation:

$$Y = m * X + C \qquad \text{(Equation 12)}$$

where

Y = measured value such as absorbance
X = concentration of the standard solution (mg/L)
m = slope of the straight line plot
C = Y intercept of the straight line plot

The slope derived from a blank and a standard is calculated and used for some procedures (e.g. ICP-AES and ICP-MS). Spreadsheets are commonly used to determine slopes, intercepts and correlation coefficients for multi-standard initial calibration (ICAL) procedures. The correlation coefficient, r (an equation is not shown in this document) measures the linearity of the data set. The correlation coefficient should be equal to or greater than 0.995 for linear regression procedures. Most instrument printouts include the coefficient of variation (r^2 or COD) and it should be greater than 0.990 for linear or non-linear regression procedures. For selected methods, it is permissible for calibration blank data (X=0) to be used as one of the data points in Equation 12. Analyst discretion is essential when calibration blank is included as one of the points in the data set. Some procedures such as the Method 175 for the analysis of methane, ethane and ethene (MEE) by the EPA Robert S. Kerr (RSK) may require the subtraction of blank result from each calibration point. However, such methods are uncommon. Sample concentrations are calculated simply by plugging in the measured value such as absorbance for the sample as Y in equation 12 and calculating X, the sample concentration. Equations used for calculating general chemistry parameters vary by the test. The AML standard operating procedure (SOP) for the applicable test should be used for additional details.

12.2 Data Verification and Review

12.2.1 Review Items

When the analysis of a sample set is completed, the results will be reviewed and evaluated to assess the validity of the data set. The review procedures are similar for most tests. The AML standard operating procedure (SOP) for the applicable test should be used for additional details. In general, review is based on the following criteria.

A. *Method Blank Evaluation* - The method blank and preparation blank results are evaluated for high readings characteristic of background contamination. If high blank values are observed, the analysis should be halted and laboratory glassware and reagents should be checked for contamination. A universally applicable blank criterion does not exist. Blank criteria vary by EPA programs (wastewater, drinking water, solid waste, Superfund, etc.), EPA method manuals (SW-846, CLP, etc.) and EPA methods. Criteria are linked to one or more of the following: IDL, MDL, MQL, regulatory limit for the various programs and/or media, associated sample result, administrative limits, etc. According to one of the appendices attached to an USACE document,

method blank levels should not exceed MDL. According to Chapter One of the SW-846 document, "Guidelines should be in place for accepting or rejecting data based on the level of contamination in the blank". Typically, a method blank should contain no greater than three times the method detection limit (MDL) or instrument detection limit (IDL), no greater than the reporting limit, or no greater than one-half of the applicable method quantitation limit (MQL). Other blank criteria such as blank result not exceeding the MQL are also commonly used in the industry. The method blank amount should not exceed one half of the applicable MQL for blank to be acceptable for analyses performed at AML.

If a target analyte/compound is detected in the method blank at levels greater than the reporting limit, associated sample data should be qualified with the "B" code and the blank amounts made available to the end user. If a target analyte, which is not a common laboratory contaminant is detected in the method blank at levels greater than one-half of the applicable MQL, corrective action should also be taken. Assigning additional qualifiers such as "U" for associated samples if the sample amount is less than five times the blank amount (less than ten times the blank amount for common contaminants) is normally the responsibility of the end user of data. The end user is also likely to take into consideration blank levels in field blanks, while assigning data qualifiers for associated data. The applicable test-specific AML SOP should be reviewed for appropriate corrective action and method-specific details. Project specific requirements should also be considered.

B. Field Blank Evaluation - Field blank results are evaluated for high reading similar to the reagent and/or method blanks. Field blanks may include equipment rinsates, blank samples exposed to field conditions, water used for drilling or decontamination and trip blanks. If high field blank readings are encountered, the procedures employed for sample collection, shipment, and laboratory analysis should be checked by data user. If method blanks and field blanks both exhibit significant background contaminations, the source of contamination may be from the laboratory. For volatile organic analyses, ambient air in the laboratory and reagents should be checked for possible sources of contamination. High field blank readings for other parameters may be due to contaminated sample bottles, contamination from equipment and chemicals used in the field, emissions from motorized equipment used in the field and sample transportation, cross-contamination due to sample leakage, or poorly sealed sample containers. The laboratory will not evaluate the results for field blank samples. Assigning qualifiers for associated samples based on field blank results is strictly performed by the end user of data and not by the laboratory. The applicable test-specific AML SOP should be reviewed for details and project specific requirements should also be considered.

C. Calibration Evaluation - The initial calibration curve (ICAL) used for sample quantitation is evaluated to determine linearity through its full range, and that sample values are within the range defined by the low and high standards of the ICAL. Linear regression may be used when procedures that assume linearity through zero (CF and RF methods) are not applicable. When linearity is not achievable, which may be the case for some analytes, sample values must be corrected for nonlinearity by deriving sample concentrations from a graph or by using an appropriate algorithm to fit a nonlinear curve to the standards. When an ICAL that was constructed previously is verified by analyzing a CCV during sample analysis, deviations from the ICAL are also noted. Samples may require reanalysis if the deviations exceed applicable QC limits. Deviations that exceed QC limits

for specific analytes in multi-analyte methods may need to be documented in the project case narrative. Assigning qualifiers for associated samples based on calibration anomalies is normally performed by the end user of the data and not by the laboratory. See applicable test-specific AML SOPs for details.

D. Laboratory Control Sample Evaluation – As described in Section 5 of this manual, a minimum of one LCS will be analyzed with each set of samples, which is called a quality control batch (QC batch). For critical projects, LCS will be analyzed in duplicate. The results of laboratory control sample (LCS), LCS duplicate (LCSD) or quality control check standard (QC) analyses are compared with true values and the percent recovery is calculated. The percent recovery value may be plotted on a control chart for the parameter determined. When the percent recovery value exceeds the warning limit (2-sigma for in-house statistical limits) for the parameter in question, no action is necessary except to notify the laboratory management. When the percent recovery value exceeds the laboratory control limit (3-sigma for in-house statistical limits), it may be indicative of more serious problems and corrective action will be necessary. Warning and control limits for LCS are determined statistically from the mean and standard deviation of the observed recoveries as described elsewhere in the AML QAP document. LCS analyte recoveries within in-house control limits are acceptable and data can be reported without qualifiers.

Ideally, each sampling and analysis project should establish its own acceptance criteria for LCS recoveries based on project data quality objectives (DQOs) and this information should be communicated to the laboratory by providing a copy of the Quality Assurance Project Plan (QAPP). Whenever available, the laboratory should use these project-specific limits in analytical reports. For some projects, project-specific limits may be incorporated by reference to administrative limits included in guidance documents such as the Department of Defense Quality Systems Manual (DoD QSM), USACE Shell document (EM 200-1-3-I.4.7.2.1), etc. These administrative or default limits for LCS recoveries are based on the method quality objectives (MQOs) process, which are more general in nature. These administrative limits (e.g. DoD QSM, Appendix DoD-D, LCS Control Limits) may be used as default criteria when more explicit project specific limits are unavailable.

The in-house statistically derived control limits should be consistent with project specific limits or default limits. If the laboratory does not meet project specific requirements, efforts must be made to improve in-house method performance for the affected analytes. Negotiations with the client to use a different method, flag data, etc. may be necessary prior to sample analysis. When project-specified limits are wider than in-house limits, it is allowable to report data meeting project-specific limits but not the in-house limits. However, efforts should be made to investigate and correct the cause of failure based on the tighter in-house limits to minimize systematic errors.

AML employs default 3-sigma limits contained in the DoD QSM (Final Revision 2, June 2002) with minor modifications as control limits. The USACE and DoD documents also specify limits for the number of analytes (Table D-1 of the DoD QSM) that may exceed the 3-sigma limits for multi-analyte methods. The allowable number of marginal exceedances is related to the number of total analytes for the method: 0 for <11 analytes, 1 for 11-30 analytes, 2 for 31-50 analytes, 3 for 51-70 analytes, 4 for 71-90 analytes, and 5 for >90 analytes. The DoD QSM also specifies marginal

exceedance (ME) limits, which are set at four times the standard deviation (4-sigma) around the mean. The ME limits should not be exceeded for the associated data to be usable as valid data.

Each LCS should be evaluated against the DoD's control limits and ME limits before being accepted. The AML LIMS stores both limits for each method and compares the observed LCS recovery against these limits. If the observed recovery is within the control limits, the recovery data are not flagged. If the recovery falls outside the control limits and it is within the ME limits, the recovery data are flagged as "ME". If the recovery falls outside the ME limits, the recovery data are flagged as "ME*". The AML LIMS also calculates the number of analytes reported for the method and the number of compounds that exceeded the control limits. These are shown at the bottom of LCS recovery reports along with the number of allowable ME analytes per DoD QSM. Corrective action should be performed if the actual number of compounds that exceeded the LCS limits is greater than the limit allowed by the DoD QSM. Corrective action may involve checking the validity of integration performed by the instrument, chromatography problems, calibration issues for the affected compound, etc. The LCS or QC check standard may need to be reanalyzed and the success of the corrective action procedure should be documented.

If reanalysis of the LCS is not successful, the QA Manager should be notified, and re-extraction and/or reanalysis of all associated samples for the affected parameters may be needed. The laboratory project manager may need to inform the client project manager, project chemist, etc. and formulate an appropriate corrective action procedure that meets project requirements. Due to the complexity associated with multi-analyte methods, applicable test-specific AML SOP should be used for detailed guidance. Assigning qualifiers for specific analytes in associated samples based on LCS recoveries is normally performed by the end user of the data and not by the laboratory, when valid data are not attainable after corrective action is performed by the laboratory.

E. Surrogate Recovery Evaluation - The results of surrogate standard determinations are compared with the true values spiked into the sample matrix prior to extraction and analysis. The percent recoveries of the surrogate standards should be determined and compared with applicable quality control (QC) limits. Applicable QC limits include those established by the laboratory (default) and those derived from specific quality assurance project plans. AML employs default QC limits contained in the DoD QSM (Final Revision 2, June 2002) with minor modifications as control limits for surrogate recoveries.

Surrogate recoveries should be within control limits for data to be acceptable for organic analyses. Due to the complexity associated with multi-analyte methods that employ multiple surrogates, applicable test-specific AML SOP should also be used for detailed guidance. Re-extraction of the affected samples (at a dilution, if necessary) should be performed if instrumental re-analysis fails to resolve the anomaly. Recoveries consistently outside the QC limits for replicate analyses of the same sample are indicative of matrix related problems that are not solvable using standard corrective action procedures.

The failure of surrogate recoveries in specific samples should be described in the laboratory case narrative. Results for all analyses that failed surrogates should be reported with QC flags (e.g. "*") for failed surrogates. If an analysis that passed surrogate requirements is available, sample results

from failed surrogate recovery runs should not be reported unless the data exhibits other more serious problems. Assigning qualifiers for associated analyte/compound results for the affected samples based on surrogate recoveries is normally performed by the end user of the data and not by the laboratory. See applicable test-specific AML SOPs for details.

F. Matrix Spike Evaluation - The observed recovery of the spike versus the theoretical spike recovery is used to determine bias in analysis for specific project samples. Matrix spike (MS) analyses are normally performed in duplicate by spiking the sample and the results of the unspiked sample analysis are used in determining spike recoveries. A clean sample, representative of the site conditions is normally used for triplicate analyses (sample, MS and MSD).

The percent MS recovery value may be plotted on a control chart for the parameter determined. When the percent recovery value exceeds the warning limit for the parameter in question, the QA Manager is notified. Laboratory personnel should be aware of statistical limits as well as administrative QC limits that are derived from government agency requirements, program requirements, quality assurance project plan requirements, etc.

MS evaluation is more complex than method blank or LCS data evaluation due to complications arising from matrix effects in addition to sample preparation and analysis errors. The DoD QSM does not contain QC limits for MS and MSD recoveries. AML has adopted the QC limits for LCS contained in the DoD QSM as QC limits for MS and MSD recoveries.

If MS recoveries exceed the applicable quality control (QC) limits, it may be indicative of either laboratory problems or matrix related problems. If the recoveries for LCS or LCSD analyzed with the sample batch are acceptable, matrix related problems are strongly suspected. The heterogeneity of grab soil samples, and potential non-representativeness of sequentially collected water samples, etc. further complicate the evaluation since it is assumed that the native concentrations are constant in triplicate analyses. In addition, concentrations of the target analytes in the sample can also far exceed spike amounts. If the native concentration of target analytes in the sample chosen for spiking is high relative to the spiking concentration, the differences in the native concentration between the unspiked sample and the spiked samples may also become very significant.

In addition to their reluctance to pay for MS and/or MSD analyses, a number of laboratory clients may not know what samples to collect for MS and MSD analyses. They may not collect sufficient samples, may not have any knowledge of the native analyte concentrations in the chosen sample, and may not have knowledge of MS spiking levels that are appropriate either for the sample or their project. For procedures with multiple target analytes, spiking levels may be inappropriate for one or more analytes, if samples that contain target analytes are chosen either in the field or by the laboratory. If no project samples are collected for MS analysis and samples from another project are used for MS/MSD analysis, the recovery data may not be representative of project samples. When a specific project sample (e.g. surface soil) is chosen, their MS recovery characteristics may be different from those of other samples (e.g. sub-surface soil) in the same batch.

If the sample contains significant amounts of target analytes/compounds, the amount spiked may not be significant relative to the endogenous amounts. If the sample chosen for MS/MSD is

inappropriate, the client should be notified. When additional payments are authorized for re-analysis, MS/MSD samples may be reextracted and/or re-analyzed after employing cleanup procedures (if applicable), dilution techniques to minimize matrix interference, or using other project samples that appear to be clean, when sufficient quantities are available. According to USACE, spike amounts should be as much as five times sample concentrations to minimize the effect of sample concentration errors, and spikes at 25-100% of the endogenous amount could be too low to provide more meaningful recoveries.

If the samples are not homogenous, corrections applied for endogenous amounts while calculating MS recoveries may be in error. The MS recoveries will be meaningless if the amount spiked is less than 25% of the endogenous amounts even for inorganic analyses. Recoveries within control limits may not always be achievable due to differences within aqueous (drinking water, groundwater, wastewater, etc.) and solid (clay, sand, garden soil, sediment, solid waste, etc.) matrix characteristics. Re-spike the sample at a higher level (e.g., at approximately two to four times the sample concentration), then reanalyze the sample based on project-specific requirements. It is the responsibility of the client at all times to select appropriate samples for MS analysis, make additional payments for additional analyses that may be required, and to offer necessary guidance as needed to meet project data quality objectives.

Matrix spike recoveries are not commonly used as batch QC since the sample selected for MS/MSD analyses may not be from the site, and it may not be representative of the associated samples, even if it is from the site. For example, surface soil samples, which typically have high organic content may exhibit markedly different MS recovery characteristics from those collected at the same geographic location but at other depths. The MS recovery characteristics of each sample may differ from one another. An appropriate corrective action dealing with MS recovery anomalies is, therefore, very difficult. The sample set may be reanalyzed the same way as described for the laboratory control sample analysis if matrix problems are ruled out. Re-extraction and re-analysis of the associated samples may be necessary as a corrective action of last resort only and only if such requirements are project-specific. Assigning qualifiers for associated samples based on MS recoveries is normally performed by the end user of the data and not by the laboratory. See applicable test-specific AML SOPs for additional details. The QA Director and Project Manager should be consulted to determine the appropriate course of corrective action.

G. Duplicate Sample Evaluation - Duplicate sample analysis for the sample set is used to determine the precision of the analytical method for the sample matrix. The duplicate results are used to calculate the precision as defined by relative percent difference (RPD) between duplicate measurements. The RPDs should be plotted on control charts. If the RPD exceeds the warning limit, the QA Manager is notified. If the RPD value exceeds the control limit, the sample set may be reanalyzed for the parameter in question if the matrix is determined to be homogenous. Laboratory personnel should be aware of statistical limits as well as administrative QC limits that are derived from government agency requirements, program requirements, quality assurance project plan requirements, etc.

If other available QC parameters (surrogates, percent recoveries, RPDs for LCS duplicates, etc.) are in control, matrix inhomogeneity is likely to be the source of the anomaly and the sample data may

be reported with QC flags alerting the RPD outlier. Since the sample selected for the duplicate analysis (includes MS/MSD analysis) may or may not have been homogenized in the field, assigning qualifiers for associated samples based on RPD should be performed by the end user of data. See applicable test-specific AML SOPs for additional details.

H. Matrix Spike Duplicate Evaluation – These results, when available, are treated in the same manner as a duplicate result for the purposes of evaluating the precision of the analytical method for the group of sample being analyzed. Sample concentrations for MS and MSD samples (not percent recoveries) should be used to calculate RPDs except for procedures for which sample amounts for laboratory analyses are not controllable (e.g. 5035 method for VOCs) by the laboratory.

I. LCS Duplicate Evaluation – These results, when available are treated in the same manner as a duplicate result for the purposes of evaluating the precision of the analytical method for the group of sample being analyzed. Sample concentrations for LCS and LCSD samples (not percent recoveries) should be used to calculate RPDs. The RPDs for LCS duplicates are not evaluated since the analysis of LCS samples in duplicate are not required.

J. Standard Reference Material Evaluation - Standard Reference Material (SRM) analyses are compared with the true values and acceptable ranges, which are provided by the SRM vendor . SRMs are analyzed less frequently compared to LCS. Values outside the acceptable ranges require corrective action to determine the source of error. All sample analyses should be halted pending this evaluation. Following correction of the problem, the SRM should be reanalyzed. The performance testing (PT) samples from past WP, WS, and RCRA programs may be used as SRMs for routine environmental analyses.

Additional data quality control is accomplished by participation in performance testing studies coordinated by the State/U.S. EPA such as the Water Pollution (WP) and Water Supply (WS) programs under the National Environmental Laboratory Accreditation Program (NELAC) or U.S. Army Corps of Engineers (USACE). Successful analysis of these types of performance evaluation samples can be used to attain laboratory certification for organic and/or inorganic analyses.

12.2.2 Review Process

Data verification begins with the processing of data. Data processing can be performed by the analyst who obtained the data or by another analyst. Verification continues with checking that the data processing has been done correctly. The second step in the verification process can be performed by either a second analyst or the Team Leader. The review process should be documented by signature (or initials) and date on the documents that are verified.

In general, data will be processed by analyst in one or more of the following ways:

- Manual computation of results directly on the data sheet
- Manual computation on calculation pages attached to the data sheets
- Input of raw data for computer processing on desk-top or notebook personal computers (PC)
- Input of data in to laboratory information management system (LIMS)
- Direct acquisition and processing of raw data by the computer performing data acquisition

If data are manually processed by an analyst, significant steps in the computation shall be provided including equations used and the source of input parameters such as response factors, dilution factors, and calibration constants. The analyst shall sign and date in ink each page of calculations. For data that are input by an analyst and processed by a computer, a copy of the input shall be kept and uniquely identified with the sample numbers and other information as needed. The samples analyzed shall be evident and the input signed and dated by the analyst. If data are directly acquired from instrumentation and processed, the analyst shall verify that the following are correct: sample numbers, calibration constants, response factors, output parameters such as units, and numerical values used for detection and quantitation limits. The analyst shall sign and date the resulting output. A checklist should be completed for each batch of client samples processed by the analyst. See AML SOPs specific for each test for details.

Data reports should be reviewed to verify that information reported by AML corresponds with processed analytical results. This is first done by the Team Leader and then by the Quality Assurance Manager, Project Manager, or the Lab Director prior to being released by the laboratory. Those who review data reports should indicate such by signature on the checklists.

12.2.3 Level I Review

The analyst will draw on his/her experience in the interpretation and use of the internal or routine quality control samples in order to gauge the quality of the analytical results for each batch of samples. The analyst's primary responsibility is for the calibration of the instrumentation (ICAL, CCVs, CCBs, etc.) and incorporation and analysis of the required quality control samples (MB, LCS, LCSD, MS, MSD, etc.). The analyst will verify the sensitivity or responses (SPCC, CCC, RF, CF, etc.) of each instrument, stability of the system, elimination of baseline interferences, etc. Additionally, the analyst will check for the absence of unusually high background due to any reagents (MB, reagent blank, ICB, CCB, etc.) used throughout the analytical process. Once the analyst has verified that the instrument has been calibrated and is responding in the appropriate manner, he/she will proceed to complete the analysis of a batch of samples.

The analyst responsibilities also include the exercising of his/her own expertise in evaluating each batch of samples and the data generated, as well as comparing data for accuracy and precision using the default laboratory quality control limit tables. The analyst will check for bias or accuracy by verifying that the spike recoveries (LCS, LCSD, MS, MSD, PDS, etc.) are within the default limits established by the laboratory for the specific project. The analyst will check for precision by verifying that the replication (LCS duplicate, MS duplicate, sample duplicate, etc.) of samples (e.g. RPD) is within the prescribed limits established by the laboratory. Any variance greater than the control limit(s) may require rejection, reanalysis, re-extraction and reanalysis or qualification of all analytical results in that specific sample batch.

The analyst will complete a checklist appropriate to the test being performed. A number of checklists are available. At a minimum, the checklist used to present raw data (Table 12.1) for Level IV reports should be employed. More detailed checklists are included in AML SOPs, which are specific to the test performed (see example checklist, Figure 12.1). If specific checklists are unavailable for a test, an example Level I checklist shown in Table 12.2 may be used for each batch of client data submitted by the analyst to his/her supervisor along with a brief statement on the acceptability of the data.

Other means of documenting review may also be acceptable. The analyst may have no direct responsibility for rejection of any set of data, unless authorized otherwise by the respective supervisor.

12.2.4 Level II Review

The Level II review may be provided by a second analyst, a Team Leader or a supervisor who may be responsible for reviewing data generated by analysts within their section. The Level II review will be technically and statistically more rigorous in nature. With regard to surrogate recoveries, the Team Leader will compare recoveries for other tests run on the same sample to determine matrix related problems. The Team Leader will check calculations at the frequency listed in the Level II checklist (Table 12.3) and document any anomalies either on the test-specific checklists used by the analyst, Level II checklist shown in this document, or directly on the raw data submitted by the analyst. Other means of documenting review may also be acceptable. The Team Leader will order reanalysis as needed but, he will have no direct responsibility for rejection of any set of data that meet most but not all acceptance criteria.

12.2.5 Level III Review

Level III review will be performed by the Operations Manager, Project Manager, Quality Assurance Director or the Laboratory Director, who will have the authority and/or responsibility to approve, reject or qualify data reported to clients. Although the same accuracy charts used by the Analyst or Team Leader will be used for data assessment, the controlling factor for the acceptability or rejection of data will take into account the overall needs of the project, which may include project-specific data quality objectives, client preferences, turnaround times, etc.. The Level III review will also include a check for project completeness.

Following the Level III review, the Level III reviewer or his designee may provide specific analysts with advance notice of the potential of any system being declared out of control. An attempt will be made to determine the cause of the deviation of the analytical process. When the analyses are complete, the information recorded in the bench books is reviewed by the Analyst and the Team Leader before it is logged onto the computer for report generation. The final report is then printed and used as part of the Level III review. The revised report, following this Level III review is then printed, signed by the Level III reviewer or his/her designee before they are authorized for release to the client. Level III review comments are documented directly on test-specific checklists, custom checklists generated for each AML work order or a generic checklist similar to that shown in Table 12.4. Other means of documenting review may also be acceptable.

12.3 Recording of Data

Raw data is recorded in bound books and/or by instrument printout. This includes such information as standard curves, matrix spikes, duplicates, reagent blanks, calculations and any notations concerning a given analysis. Preventive maintenance information is kept separate from the analyst's logbook and with the assigned instrument. Raw data associated with each client report will be archived in a separate folder and kept on facility for a minimum of three years after completion of the project. See applicable AML SOP regarding data storage and retrieval procedures.

12.4 Reporting of Data

The appearance of the report will be consistent with client requirements. At a minimum, the AML report will contain the following information on Level II reports:
Cover Letter
Sample identification number assigned by the sample collector;
Laboratory sample identifier
Date of sample collection
Date (and time, if required) extraction and/or analysis was performed
Initials of the analyst (if required)
Analytical methods employed
Quality Control (QC) batch number (when required)
Results of analyses grouped by sample and reported by parameter
Units as set forth in the referenced method or in client specifications
Reporting limits (RL)
Method detection limits (MDL) and method quantitation limits (MQL), when required
Copy of the original chain of custody form

For Level III reporting, a quality assurance summary report will also be provided which will include the following for each parameter and each QC batch:
Case narrative
Results of sample analyses grouped by parameter and matrix
Quality control data grouped by parameter and matrix
- Method blank results
- Surrogate recovery results and summary with flags for QC outliers
- Laboratory control sample (LCS) and LCS duplicate (LCSD) results (if performed)
- Matrix spike (MS) and matrix spike duplicate (MSD) results (when available)
- LCS and LCSD (if performed) recoveries, QC or ME limits and flags for outliers
- MS and MSD (when available) recoveries, QC or ME limits and flags for outliers
- Summary of laboratory duplicate results (if performed)
- Summary of serial dilution (SD) test results for metals analysis
- Summary of post digestion spike (PDS) test results for metals analysis
- Summary of %D for SD results (when applicable)
- Summary of RPDs for laboratory duplicates (when applicable)
- Summary of PDS recoveries, QC limits and flags for outliers

Level IV reports (see EPA Contract Laboratory Program Guidelines for detail) will generally contain associated raw data in addition to the Level III reports. Level IV reports will also contain additional analytical information, which are test-specific as described below:
Instrument tune summary and associated raw data for GC/MS analyses
Internal standard areas and retention time summary
Initial calibration summary and associated raw data for all parameters
Calibration verification summary and associated raw data for all parameters
Sample specific work sheets for GC/HPLC analyses
Documentation of IDL, linear range, etc. for metals analyses
Copies of benchsheets used to document sample preparations
Copies of logbook pages containing analytical information

Copies of discussion items between the laboratory and client personnel
Copies of traffic reports
Other miscellaneous items (project-specific)

See applicable AML SOPs for example reports, keeping in mind that reports may vary from client to client and from project to project.

Analytical Management Laboratories

Table 12.1 - GENERIC CHECKLIST

(allowable for completion by the analyst in lieu of other checklists)

(can be modified and customized for specific parameters)

_____ Form-I for each sample

_____ Form-IV - QC Association Form with date and initials

_____ QC Summary (Form-I for MB, LCS/LCSD, MS/MSD)

_____ Form-II for surrogate recoveries (when applicable)

_____ Form-III or equivalent for Spike Recoveries (LCS/LCSD, MS/MSD, PDS)

_____ Raw data for project samples

_____ Raw data for QC samples

_____ Initial Calibration (ICAL) Summary and Raw data CCVs and CCBs – Summary and Raw Data

_____ Sample Preparation sheet (copy) and LIMS printouts

_____ _____

Date **Analyst Initials or Signature**

Analytical Management Laboratories

Table 12.2 - GENERIC LEVEL I REVIEW CHECKLIST

(recommended to be completed by the analyst in lieu of other checklists, if unavailable)

Parameter: _____ AML Work Order No:_____

#	ITEM TO BE CHECKED	YES	NO	COMMENTS
1	Sample preparation information correct and complete?			
2	Samples extracted and analyzed within technical holding times?			
3	Instrumental analysis information correct and complete?			
4	Instruments in good condition and in acceptable tune?			
5	Initial calibration acceptable?			
6	Continuing calibrations acceptable?			
7	Correct methods/SOPs followed?			
8	Sample analytical results including units correct and complete for all analyses including reruns?			
9	Blank free of analytes?			
10	LCS and/or LCSD recoveries within QC limits?			
11	Surrogate recoveries for all analyses within QC limits?			
12	MS and MSD recoveries within QC limits?			
13	RPDs for LCS/LCSSD within QC limits?			
14	RPDs for MS/MSD within QC limits?			
15	RPDs for laboratory duplicates within QC limits?			
16	Documentation complete?			
17	Special sample preparation and analysis requirements, if any, met?			

General comments, if any:			
_____	_____	_____	_____
Signature of Analyst	Date	Signature of Supervisor	Date

Analytical Management Laboratories

Table 12.3 - GENERIC LEVEL II REVIEW CHECKLIST

(recommended to be completed by the Team Leader in lieu of other checklists, if unavailable)

Section: _____ **Project No: _____**

For all tests performed by the section on samples from this SDG:

#	ITEM TO BE CHECKED	YES	NO	COMMENTS
1	Correct methods/SOPs followed?			
2	Calibration data scientifically sound, appropriate to the method and completely documented?			
3	QC (blanks, LCS, LCSD, MS, MSD, surrogate, matrix dup. Results /recoveries /RPDs) results within QC limits or guidelines?			
4	Qualitative identification of analytes correct?			
5	Calculations on 10 to 20% of raw data including calibrations and QC samples free of errors?			
6	Additional data checked including the entire data set, if necessary, if errors were detected?			
7	Quantitative results after these checks correct including units?			
8	Documentation complete and correct, including documentation of anomalies in holding times, sample preparation and analysis?			
9	Data package complete and ready for submittal?			
10	Data qualified, if necessary, and ready for incorporation into the final report and/or LIMS?			

General comments, if any:
_____ _____ _____ Signature of Team Leader Date Signature of Project Manager Date

Analytical Management Laboratories

Table 12.4 - GENERIC LEVEL III REVIEW CHECKLIST

(recommended to be completed by the QA Officer, Project Manager or Lab Director in lieu of other checklists)

Department: _____ **Project No: _____**

For all tests performed by the laboratory on samples from this SDG:

#	ITEM TO BE CHECKED	YES	NO	COMMENTS
1	A. Deficiencies identified in Level I and II reviews corrected? B. All the required analyses performed?			
2	Test data for samples in this WO consistent, free of anomalies and free of errors including unit errors?			
3	Data in compliance with client contractual requirements including special requirements, if any?			
4	Case narrative, if necessary, prepared and reviewed?			
5	A. Electronic Data Deliverables (EDD) required? B. EDD in the required format?			
6	Data in EDD match hardcopy data?			
7	Sample disposal form completed for this SDG in accordance with laboratory policy and client specifications?			
8	Task order exists for billing the analysis?			
9	Invoices completed for this SDG in accordance with contract requirements, including adjustments for non-compliance?			

General comments, if any:

_____ _____

Signature of person signing-off on report Date

CLERICAL CHECKLIST

Date(s) partial reports were faxed or emailed:	
Date final report was faxed or emailed:	
Date final report was sent and the courier used (US Mail, FedEx, Airborne etc.)	
Date invoice was sent and invoice number:	
Signature of the Administrative Asst.:	Date:

13.0 CORRECTIVE ACTION

Corrective action will be initiated whenever the ongoing quality control/quality assurance program identifies deficiencies that will require corrective action. Deficiencies may exist when QC parameters are outside the control or acceptance limits or when data are determined to be questionable. For routine operational problems, an immediate short-term corrective action is appropriate. For example, reanalysis of samples with low surrogate recoveries, repairing and/or retuning an instrument that is working improperly are short-term corrective actions. Long-term corrective actions are aimed at eliminating the source of systematic errors in sampling and analysis, which may be manifested by recurring poor performance in audit sample analysis. Corrective action appropriate to each situation is addressed as shown below:

13.1 Day-to-day Quality Control parameters

One or more routine QC parameters associated with environmental sample analysis such as sample holding times, instrument tune, blank, surrogate recoveries, initial calibration, continuing calibration, laboratory control sample, QC check standard, laboratory duplicate, matrix spike recovery, and precision in matrix spike duplicate recoveries may be outside control limits. In such cases, corrective action is initiated by the analyst (staff chemist or technician) and reviewed by the team leader. If the review, which may include checking procedures, calculations, instrument operating conditions, reagents, standard solutions, etc. does not correct the problem(s), analysis is repeated provided sufficient sample extract, sample, and time are available.

The AML data reduction, review and verification procedures described in Section 12 of this document and the referenced test specific SOPs may necessitate corrective action(s) to achieve project specific data quality objectives. The test-specific AML SOPs contain detailed guidelines for corrective action, which should be followed. Summary guidelines contained in Table 13.2 should be followed, if the SOP does not contain sufficient guidelines.

If data are submitted in cases where QC parameters are not in compliance, data qualification may be necessary. For example, cases such as those involving the presence of analyte(s) in associated method blank (B code), sample amounts below (J code) or above (E code) calibration limits, etc., the laboratory will qualify sample results appropriately as shown in parentheses. In other cases, such as those involving calibration, LCS recoveries, MS recoveries, RPD, etc. the case narrative should include recommendations for data qualifications for the end user, who may perform an independent review.

Table 13.2 contains a summary of corrective action procedures to be followed for each type QC deviation episode. Laboratory personnel should refer to method specific AML standard operating procedures (SOPs) for more detailed guidance that are more specific.

13.2 Performance Evaluation Samples

Blind performance testing or evaluation (PT or PE) samples are periodically analyzed by the laboratory to support various accreditation or certification programs run by various organizations such as EPA, USACE, State of Kansas (e.g. NELAP), etc. Deficiencies identified in the performance reports should be addressed by the Analyst, Team Leader, and the management staff. The response should preferably be in writing and it should indicate where possible the cause of the deficiency and actions taken to correct the problem and to prevent recurrence. Copies of the responses should be provided to the laboratory director (LD) and the quality assurance director (QAD).

13.3 On-Site System Performance Audits

Generally, these are conducted for the purpose of supporting external QA programs. Most audits end with an "exit briefing", in which the laboratory director and/or the QAD are briefed on the problems, which are followed with written reports by the auditor. The QAD is responsible for responding to these verbal and written reports and for recommending corrective actions to be undertaken by the laboratory. The Operations Manager and the laboratory staff are responsible for implementing corrective action proposed by the QAD.

13.4 Special Concerns

Analytical Management Laboratories will abide by any corrective action deemed necessary by EPA, state or other pertinent agency as a matter of policy.

13.5 Communication and Documentation of Corrective Action

When situations requiring corrective action(s) occur, the situation is communicated either verbally or in writing as appropriate. Email can also be used as appropriate.

For minor day-to-day (short term problems) problems, verbal communication by the analyst to the team leader and a short note on the instrument notebook, bench book or other appropriate document is sufficient in most instances. For projects that require detailed written documentation, written corrective action forms (Figure 13.1) should be initiated by the analyst. This documentation should describe the problem and the proposed solution, which can be proposed by the analyst and/or the team leader. The responsibility for implementation is normally assigned to the analyst with the responsibility for follow-up resting with the team leader. The QAD will be in the loop to provide additional suggestions. The QAD may include the laboratory project manager (if assigned) and the client (e.g. project chemist at the A/E firm) in project related discussions, which may include modifications to the proposed corrective action.

For persistent or systematic problems and for problems identified by external auditors (long term problems), QAD and/or the LD will initiate appropriate corrective action. Proposed solutions will be formulated by a team that involves senior laboratory personnel (e.g. Operations Manager (OM), Information Technology Manager (ITM), Team Leaders, etc.), QAD and LD. Long-term problems require documentation on the Corrective Action Report Form (Figure 13.1), which should be submitted to the OM. The responsibility for implementation will rest with the operations manager, which may be delegated as necessary to team leaders. The responsibility for follow-up will rest with the QAD.

FIGURE 13.1

AML CORRECTIVE ACTION REPORT

DATE:	ORIGINATOR:	CAR No.:

NATURE OF THE PROBLEM:

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RECOMMENDED ACTION:

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IMPLEMENTATION OF REQUIRED ACTION:

Responsibility for implementation assigned to: _____ Due date: _____
Responsibility for follow-up assigned to: _____ Due date: _____
Copies of CAR sent to: ☐ QAO ☐ Lab Director ☐ Team Leader ☐ Other: _____

CORRECTIVE ACTION FOLLOW-UP COMMENTS:

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QAM: _____ Date: _____ Team Leader: _____ Date: _____

TABLE 13.1
SUMMARY OF CORRECTIVE ACTION PROCEDURES

QC ACTIVITY	**ACCEPTANCE CRITERIA**	**RECOMMENDED CORRECTIVE ACTION**
Tuning Procedures GC/MS	Per test-specific SOP or EPA Method	Per SOP – retune, clean, repair and or adjust. Do not analyze samples until criteria are met.
Calibration Procedures Initial Calibration (ICAL) Continuing Calibrations (ICV and CCVs)	Per test-specific SOP %RSD for RF or CF Correlation coefficient for regression methods %D for CCVs	Per SOP – Check calculations Check the instrument Check the standard(s) Prepare fresh standard(s) Repeat ICAL and/or CCV Describe in case narrative
Method blanks (MB)	Target Analyte amounts less than one half of the applicable MQL	Per SOP Identify/correct problem Reanalyze blank and associated samples. Describe in case narrative
Surrogate or system monitoring compound (SMC) recoveries GC/MS analysis	Per test-specific SOP All SMCs in for VOCs 2 BN SMCs in for SVOCs 2 Acid SMCs in for SVOCs	Per SOP. Identify/Correct problem Reanalyze sample extract Re-extract/Re-analyze sample Describe in case narrative.
Surrogate or system monitoring compound (SMC) recoveries GC or HPLC analysis	Per test-specific SOP All SMCs within laboratory QC limits	Per SOP. Identify/Correct problem Reanalyze sample extract Re-extract/Re-analyze sample Describe in case narrative.
Laboratory Control Samples (LCS and or LCSD) or QC Check Samples	Per test-specific SOP %Recoveries within QC limits per DoD QSM Limits for marginal exceedances (ME)	Per SOP-Check calculations Reanalyze LCS/LCSD Re-extract associated samples, if possible. Describe in case narrative
Matrix Spike Recoveries (MS and MSD)	Per test-specific SOP %Recoveries within QC limits per DoD QSM Limits for marginal exceedances (ME)	Per SOP-Check calculations Check for matrix interference Check sample & spike levels Check LCS/LCSD recovery Describe in case narrative
LCS Duplicates Matrix Spike Duplicates Laboratory Duplicates	Per test-specific SOP RPDs within QC limits for MS and sample duplicates	Per SOP – Check sample for Homogeneity/spike levels Describe in case narrative

14.0 PERFORMANCE AND SYSTEM AUDITS

Successful and effective implementation of the quality assurance (QA) program requires development, routine checks and periodic assessment of the degree of its implementation and the effectiveness of the program in controlling quality of the analytical process. Periodic assessment, including an annual management review is performed by conducting audits designed to provide this critical means to overview and assess the procedures to ensure that they meet the objectives and goals of the quality assurance program plan. The annual review should take into account reports from managerial and supervisory personnel, the outcome of recent internal audits, assessments by external bodies (e.g. KDHE inspections, USACE inspections), the results of proficiency tests, any changes in the volume and type of work undertaken, corrective actions and other relevant factors. Overall, the audit system is expected to identify strengths and more importantly areas of weakness so that appropriate corrective actions may be taken to alleviate problems. Other objectives of the audits include the following: to facilitate the initiation of changes aimed at enhancing the QA program; to serve as a vehicle for providing technical assistance, as appropriate; to enhance awareness and understanding of QA and quality control (QC) policies and procedures; and to provide a measurement of the effectiveness of QC in assuring the quality of data. All audits and review findings and any corrective actions that arise from them should be documented in appropriate AML forms and notes. The laboratory management has the responsibility for implementing corrective actions within an agreed time frame. Analytical Management Laboratories (AML) standard operating procedure (SOP) on this subject should provide additional details not contained in this Section.

14.1 Performance Audits

Audits can be internal or external. Results from these external and internal audits are used to continually monitor lab performance and correct any out-of-control situations.

Internal performance audits will be planned and executed by the Quality Assurance Manager (QAD) annually. These audits may consist of a combination of blind quality control check samples, standard reference materials (SRMs), known standards, blind spike samples or past or current external performance testing (PT) samples such as water supply (WS) and water pollution (WP) from Analytical Products Group (APG) or Environmental Resource Associates (ERA). Audit samples are treated as actual samples and are logged in as actual client projects. Performance audits will cover targeted service areas.

Upon completion of the audits, a report containing the reported results, the true value and control limits will be sent to the Team Leaders and the Laboratory Director. The report will also outline areas of concern and it will prompt review and implementation of appropriate corrective action to alleviate problem areas. The report and any response received from the Team Leaders will become a part of the QA reports to the management.

External performance audits are performed semiannually. AML participates in the following external audit programs: Environmental Protection Agency (EPA) water pollution (WP) program, water supply (WS) and soil (RCRA) programs; and U.S. Army Corps of Engineers laboratory (USACE) environmental laboratory validation program. The QAD, Team Leaders, Operations Manager, and the Laboratory Director will review the results and then inspect all relevant documentation for deficiencies or inconsistencies. After every performance audit, the QAD will issue a report to the certifying agency forwarding a copy to the Laboratory Director, outlining the corrective action to be taken for unacceptable results.

14.2 Internal Technical Audits

Internal technical audits are conducted periodically to assess laboratory performance in stated service areas. The purpose of the internal audit is to identify strengths and weaknesses and to address weakness in order to improve laboratory performance. Quarterly audits may cover one or more technical areas. Not all areas may be covered in each audit. The audit should cover the following: initial demonstration of performance for new testing services, method detection limits, performance data for laboratory control samples, existence of written procedures that could be in the form of methods and/or SOPs, and adherence to test procedures. Test-specific checklists (e.g. Figure 12.1) or other appropriate forms (e.g. forms included on AML SOP on audits) may be used for performing and documenting internal audits. The audit will determine whether quality control standards such as blanks, LCS, matrix spikes, duplicates, etc., are incorporated with sample analytical runs at the proper frequency. Electronic audit functions available in the instrumentation will also be used to check for proper manual integration procedures, adherence to calibration procedures as described in the AML SOPs and conformance with the ethics policies.

14.3 **System Audits**

System audits are performed to determine if the entire operation is performing in accordance with the policies of the AML including adherence to the QA plans, safety plans and other plans governing the operation. System audits can be internal or external.

Internal system audits may be planned and executed either by the QAD or by an audit team once a year. The overall laboratory QA/QC program will be monitored at this time. Figure 14.1 is an example of the checklist used by the QAD to ensure that all major areas of the laboratory are included in the audit. The technical audit will determine whether quality control standards such as blanks, LCS, matrix spikes, duplicates, etc., are incorporated with sample analytical runs at the proper frequency. Systems audits will verify compliance with the laboratory's quality systems, which are based on the NELAC Quality System and client specified requirements such those contained in the Department of Defense Quality Systems Manual. Methods for responding to complaints, sample acceptance policies, and sample tracking procedures will also be examined as part of the systems audit. The audits will involve interviews of the analysts designed to improve and implement more effective procedures, evaluate training needs, and to address resource requirements. At the conclusion of the audit, the QAD will provide a written report to the Team Leaders, Operations Manager and Laboratory Director for consideration of any recommended corrective action. Follow-up audit activities will be used to verify and record the implementation and effectiveness of the corrective action taken.

If the internal technical and/or system audits reveal serious problems that might have resulted in serious errors for the laboratory results, the client should be contacted and revised reports may need to be issued if the errors are correctable. Client notification should be completed generally within 30 days after the errors were discovered. They should be addressed sooner depending on the magnitude of the problem and its impact on the defensibility and use of data.

External audits are performed annually or at a frequency designated by the certifying agencies and clients. The laboratory is audited annually or at a frequency determined by the Kansas Department of Health and Environment (KDHE) under the National Environmental Laboratory Accreditation Conference (NELAC). The QAD and/or the Laboratory Director may coordinate external audits. Field system audits may be performed periodically by various federal, state and client groups.

Figure 14.1

LABORATORY SYSTEMS AUDIT CHECKLIST
Analytical Management Laboratories

Quality Assurance Laboratory Procedures

General QA Procedures
_____Does laboratory has a general quality assurance plan?
_____Does laboratory has written SOPs to cover routine laboratory practices?
_____Does laboratory participates in any performance audit or accreditation programs?
_____Has corrective action been documented for unsatisfactory PE performance?

Chain-of-Custody
_____Is COC present?
_____Does sample information agree with COC?
_____Is signature and date and time of receipt on COC?

Sample Login
_____Was project logged in correctly and promptly?
_____Was all necessary login information included?
_____Was sample condition and preservation noted?
_____Were samples assigned unique numbers and were internal and client number both included on login record?
_____Were sample containers and lids both clearly labeled?

Sample Storage
_____Were samples and sample extracts properly stored during the project?
_____Is sample storage area clean and orderly?

Labware and Sample Container Preparation
_____Does lab maintains a distilled/deionized water system?
_____Was labware preparation adequate?
_____Does lab follow written SOPs for labware preparation?
_____Were sample containers supplied by the laboratory?
_____Were sample containers purchased pre-cleaned or cleaned according to applicable protocols?
_____Are sample containers sent to client pre-preserved and is this documented?

Sample Preparation
_____Do sample preparation methods follow SOPs?
_____Is sample prep area clean and free of clutter?

Solvents, Chemicals, and Standards
_____Were the standards correctly prepared?
_____Was the standards preparation correctly documented?
_____Are standards and chemicals labeled and dated correctly?

Figure 14.1 (continued)

LABORATORY SYSTEMS AUDIT CHECKLIST
Analytical Management Laboratories

Equipment Calibration and Maintenance
_____Was necessary instrument calibration met?
_____Was instrument calibration documented?
_____Were calibration logbooks for thermometers, refrigerators, balances, etc. complete?
_____Manual integration procedures when performed meet SOP and ethics requirements?
_____Are instrument maintenance logs up-to-date?
_____Is routine maintenance up-to-date?
_____Was equipment in working order at the time of the analysis?
_____Is equipment adequate for the method used?
_____Electronic audit functions reveal problems?

Sample Analysis
_____Did sample analyses follow the specified method?
_____Were any deviations form the specified method documented?
_____Were the sample holding times observed?

QC Sample Analysis
_____Were the required QC samples analyzed at the proper frequency?
_____Were blank samples analyzed at the proper frequency?
_____Are QC data retrievable for all analytical results?

Raw Data Storage
_____Is the raw data properly stored and easily retrieved?
_____Is the raw data legible and well preserved?

Quality Control Data
_____Were the QC objectives met for this sample?
_____Is QC data within the desired control limits?
_____Were out-of-control samples documented?
_____Was any corrective action properly documented?

Client Report
_____Did report go through the proper validation process?
_____Were all calculations correct?
_____Was the report completed on time?
_____Were units, Detection limits, etc. correctly reported?
_____Was there any client feedback on this report?

Report Storage and Archiving
_____Is raw data for this report correctly stored or archived?
_____Was the client report for this project correctly stored or archived?

Figure 14.1 (continued)

LABORATORY SYSTEMS AUDIT CHECKLIST
Analytical Management Laboratories

Administration

Client Invoice
_____Was the project invoiced in a timely manner?
_____Was the invoice correct and complete?

Policies and Procedures
_____Were Team Leaders familiar with company policies?
_____Were Team Leaders familiar with company procedures?
_____Were Team Leaders familiar with company benefit plans?
_____Were Team Leaders familiar with company goals?
_____Were employees familiar with company policies and procedures?
_____Were employees familiar with company benefit plans and its goals?

Laboratory Safety
_____Does the laboratory have a chemical hygiene plan?
_____Are MSDS's available?
_____Does the laboratory have a safety designee or safety committee?
_____Have all lab employees been trained in lab safety practices?
_____Are written records kept concerning lab safety training?
_____Does the laboratory perform safety audits periodically?
_____Are fume hoods checked periodically?
_____Are there written records for fume hood checks?
_____Are eye-washes available?
_____Are fire extinguishers present?
_____Are lab coats, gloves, and eye protection utilized where appropriate?
_____Are there any obvious safety hazards?

Sample and Waste Disposal
Procedures exist for handling and disposal of lab samples and waste?
Are waste materials properly secured and stored?
How are waste materials disposed?

Employee Satisfaction
Did the employees receive adequate training to perform assigned tasks?
Were sufficient resources provided to complete tasks on time?
Annual performance evaluation performed?
Does a mechanism exist for addressing employee grievances?

Client Satisfaction
_____Did the client receive the project results on time?
_____Were we responsive to the client needs?
_____Suggestions from clients to improve our services obtained?

15.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

AML's quality assurance approach includes an open and continuous dialog between all parties involved in the environmental data collection process, which includes the following: field and laboratory teams; team leaders and staff assigned to each team; AML and client project managers; laboratory director; vendors; and the quality assurance director. This continuous process provides the responsiveness necessary for AML to be able to monitor its operations and fulfill its mission. In addition to this continuous communication, the QA Director is responsible for preparing periodic QA reports for the Laboratory Director. See AML SOP on audits for more details. The report should include the following:

- One-page executive summary detailing uncompleted corrective actions which are more than 30 days old.
- Significant QA problems which may impact data quality or production and recommended solutions.
- Summary of external system audits and any responses/corrective action to problems noted.
- Summary of internal system audits and any responses/corrective action to problems noted.
- Summary of all external performance audit results and corrective actions.
- Summary of all internal performance audit results and corrective actions.
- Summary of precision and accuracy data for preceding six months.
- Summary of recommended changes to the standard operating procedures.

16.0 PERSONNEL QUALIFICATIONS

The organization of Analytical Management Laboratories has been described in Section 4 of this document. A list of key employees, their credentials and their role in the organization are included in this section. Clear lines of authority, duties and responsibilities consistent with the training and experience of staff, and a clear mission statement are the foundations of AML to provide our clients with services that are efficient and reliable. The qualifications of our personnel are summarized below. Resumes of key personnel are included in this document. Detailed resumes of the AML staff can be found in other documents such as the AML Statement of Qualifications (SOQ).

Mr. Bill Said is the Laboratory Director with over nineteen (19) years of laboratory experience both in industry and government. He is experienced in business development and in the operation and maintenance of common environmental laboratory equipment such as gas chromatograph-mass spectrometers (GC/MS), gas chromatographs (GC), high performance liquid chromatographs (HPLC), inductively coupled plasma atomic emission spectrometers (ICP-AES), graphite furnace (GF) atomic absorption (AA) spectrometers (GF-AA), cold vapor AA mercury analyzers (CV-AA), and total organic carbon (TOC) analyzers. He is also experienced in performing environmental sample analysis for various Environmental Protection Agency (EPA), U.S. Army Corps of Engineers (USACE), and other Department of Defense (DoD) programs.

Dr. Tenkasi Viswanathan is the Quality Assurance and Technical Director with over thirty-four (34) years of laboratory experience in both industry and government. He is experienced in the operation of common environmental laboratory equipment such as gas chromatograph-mass spectrometers (GC/MS), gas chromatographs (GC), and high performance liquid chromatographs (HPLC). He is also experienced in laboratory management, quality control procedures, quality assurance practices, program development, government contracts management, and in business development. He is experienced in preparing work plans, sampling and analysis plans (SAP), quality assurance project plans (QAPP), quality control summary reports (QCSR) and related documents. He is responsible for the development of policies and procedures for environmental laboratory operation and for quality management. He also performs report review and Level I/II/III review of environmental analytical data.

Mr. Kendall Lindquist is the Operations Manager and Principal Organic Analytical Chemist with over thirteen (13) years of laboratory experience. He is experienced in laboratory management, and in the operation and maintenance of common environmental laboratory equipment such as gas chromatograph-mass spectrometers (GC/MS), gas chromatographs (GC), and cold vapor AA mercury analyzers (CV-AA). He operates the mass spectrometers and gas chromatographs. He schedules sample analysis for AML projects and supervises the entire laboratory operation. He also performs report review. He also performs Level I/II/III review of environmental analytical data and report review.

Mr. Robert Hobson is a Team Leader and Organic Analyst with over twelve (12) years of laboratory experience. He has been trained in laboratory management and he is experienced in the

operation and maintenance of common environmental laboratory equipment such as gas chromatograph-mass spectrometers (GC/MS), gas chromatographs (GC), and high performance liquid chromatographs (HPLC). He is dedicated to performing instrumental analysis for explosives and PAHs using HPLC, coordinating organic sample preparation and for providing training and/or assistance for GC analyses. He also performs Level I/II/III review of environmental analytical data.

Mr. Daniel Krugjohn is the information technology and data management Team Leader with three (3) years of experience. He is experienced in data processing, laboratory information management system (LIMS) management, and in interfacing the LIMS with common environmental laboratory equipment such as GC/MS, GC, and inductively coupled plasma atomic emission (ICP-AES) and mass spectrometer (ICP-MS) systems. He is responsible for the operation and maintenance of the LIMS, sample management, report generation and the preparation of electronic data deliverables (EDDs). He maintains the AML internet, email, and other electronic communication systems and develops custom software for data processing and reporting.

Mr. Joseph Preheim is a Team Leader and Inorganic Analyst with fifteen (15) years of laboratory experience. He is experienced in the operation and maintenance of the ICP-AES, ICP-Mass Spectrometer (ICP-MS), and GFAA instruments. He performs instrumental analysis and coordinates inorganic sample preparation in the metals area. He also performs Level I/II review of metals data.

Mr. Robb Said is a Team Leader and Organic Analyst with fifteen (15) years of general business experience and six years of environmental sampling and/or analytical experience. He is responsible for managing the laboratory's procurement activities, sample management, field services, sample pickup, waste management, and for assisting in laboratory operations.

Mr. James Morgan is an Organic Analyst with twenty (20) years of laboratory experience. He is responsible for the operation of mass spectrometers and gas chromatographs. He performs analysis for VOCs, natural gas, and TPH-volatiles. He also performs level I/II review of organic analytical data.

Mr. Mani Ananth is an Inorganic Analyst with thirty-seven (37) years of laboratory experience. He is experienced in the operation and maintenance of the Inductively Coupled Plasma Atomic Emission spectrometer (ICP-AES). He is also a back-up analysts for operation of the ICP-MS. He is responsible for level I/II review of inorganic analytical data.

Mr. Nurani Ramakrishnan is an Organic Analyst with ten (10) years of laboratory experience. He is responsible for the operation of mass spectrometers and gas chromatographs. He performs analysis for SVOCs and TPH-extractables. He also performs level I/II review of organic analytical data.

Mr. Joseph Ahlvin is an Organic Analyst with three (3) years of laboratory experience. He is responsible for the operation of gas chromatographs. He performs analysis for organochlorine pesticides, herbicides, PCBs, and GC-Volatiles. He also performs level I/II review of organic analytical data.

Ms. Karen Zimmerly is an Inorganic Analyst and report preparation technician with over fifteen (15) years of environmental laboratory experience. She is responsible for providing assistance for CLP report preparation, operation of the ion chromatography equipment, and for performing various classical chemistry tests.

Ms. Jennifer Baumann is an Inorganic Analyst with over six (6) years of environmental laboratory experience. She is responsible for performing ion chromatographic and wet chemistry analyses.

Mr. Kevin Kennedy is a part-time inorganic Analyst/Consultant with fifteen (15) years of experience in environmental analysis. He provides technical support for metals analysis and for the operation of ICP-AES and AA equipment.

Ms. Amy Petrosino is an Inorganic Analysis Technician with five (5) years of experience. She is responsible for preparing environmental samples for ICP-AES, ICP-MS, GFAA, and CVAA analysis. She is also responsible for assisting the metals analysts in the operation of the GFAA and CVAA spectrometers.

Mr. Tariq Mehmood is an Organic Sample Preparation Technician with four (4) years of experience. He is responsible for the preparation of environmental samples for the analysis of SVOCs, pesticides, PCBs, herbicides, TPH, explosives, etc.

Ms. Rukshanda Mehmood is a part time Organic Sample Preparation Technician with one (1) year of experience. She is responsible for assisting in the preparation of environmental samples for the analysis of SVOCs, pesticides, PCBs, herbicides, TPH, explosives, etc.

Ms. Nissa Said is the Sample Custodian with three (3) years of experience. She is responsible for sample-receipt, -login, -storage, -custody and -disposal. She also prepares the coolers, bottles, labels, packaging materials, and other materials necessary for sample collection. She also provides technical support for the laboratory telephone communication system.

Ms. Kari Morgan is an Administrative Assistant with fifteen (15) years of general business experience. She is responsible for paginating, copying and scanning reports, preparation and collection of invoices, and for handling accounts payable. She also serves as the laboratory receptionist.

Bill L. Said
Laboratory Director

RELEVANT EXPERIENCE

Bill Said, Laboratory Director (from 1993) at Analytical Management Laboratories (AML) provides leadership and direction for laboratory operation, management, sales and marketing. He is one of the co-founders and owners of the laboratory and he devotes his attention to fulfilling client needs. As the Laboratory Director, he also ensures that all activities are conducted in accordance with applicable federal, state and local regulations.

Mr. Said has extensive experience in environmental analytical procedures. Prior to starting AML, he was a Senior Chemist for four years with Mantech Environmental, providing on-site technical assistance to EPA Region VII laboratory in Kansas City, Kansas. He was responsible for developing standard operating procedures for reviewing organic analytical data collected for RCRA and TCLP programs and for training personnel in the operation, and maintenance of GC/ECD, GC/MS, and other organic analytical instrumentation. He provided technical assistance for the implementation of SW-846 organic methodologies and he was a System Manager for Hewlett Packard RTE-A GC/MS data system. For two years, he also served as a Group Leader at EMS-Heritage Laboratories at Kansas City, MO, where he analyzed environmental samples for organic parameters (VOCs, SVOCs, pesticides/PCBs) in accordance with EPA Contract Laboratory Program (CLP) statement of work (SOW). He generated CLP forms and diskette deliverables, organized data package, reviewed contract compliance screen reports and performed corrective actions as required.

Mr. Said also served as a Project Manager for three years at Kansas City Analytical Services, Inc., where he managed analytical projects for various pharmaceutical companies. He supervised technical staff responsible for sample extraction of samples, GC and HPLC analysis. He also provided assistance for preparing data validation reports for the laboratory director. For two years after graduation he was a Sample Preparation Technician at ABC Laboratories, Columbia, MO. He extracted samples for analysis by senior chemists. He also prepared reagents, calibration standards, etc. and assisted in performing calculations for data reporting.

EDUCATION

B.A., Chemistry and Biology, 1985, Avila College, Kansas City, MO

PROFESSIONAL ORGANIZATION

Member, American Chemical Society
Member, KC Small Business Association

Tenkasi S. Viswanathan, Ph.D.
Quality Assurance Director

RELEVANT EXPERIENCE

Dr. Viswanathan, manager of quality assurance at AML (from 1998) provides guidance and leadership for quality control and quality management for the rapidly growing environmental laboratory. As one of the owners of the laboratory, he provides expertise in laboratory management, financial management, client development, and program development. He develops and maintains the laboratory quality assurance program, standard operating procedures, safety program, laboratory certification program and the regulatory compliance program.

Prior to joining AML as a full time QA Manager, he served for two years as a chemist at the US Army Corps of Engineers (USACE), Kansas City District. He was responsible for project development, development of contractor scope of work (SOW) and for the review and/or development of project documents such as SAP, FSP, QAPP, QCP, SSHP, etc. He was a member of remedial design teams for a number of EPA Region II, IV and VII Superfund projects, FUSRAP, IRP and FUDs projects. From 1989 to 1995, he held various management positions at PSI, Lawrence, KS serving as a Program Manager, District Manager and as a Department/Division Manager for a multi-location, full-service environmental and industrial hygiene laboratory. He had responsibilities for day to day operation, quality assurance, report review, marketing and program development for the Lawrence, KS laboratory with approx. 45 employees.

For six years from 1983 to 1989 he provided technical assistance and managed various government contracts at EPA Region VII. He started up the NSI/Mantech Region VII EPA Environmental Services Assistance Team (ESAT) in Kansas City and served as its branch manager for two years. The ESAT team provided technical support for data and document review, sample management and analytical services. As a section leader for E&E and Jacobs Engineering Group's laboratory Technical Assistance Teams (TAT), he also supervised/performed data review and operated gas chromatographs and mass spectrometers (low and high resolution) to perform environmental analysis. He developed TCDD and PCDD/PCDF analytical methods for use at EPA Region VII. From 1975 to 1983 he held various research and teaching appointments at University of Kansas Medical Center, Simon Fraser University (Canada), Massachusetts Institute of Technology and University of Virginia. He published over 20 articles in peer-reviewed journals covering NMR and mass spectrometry.

EDUCATION

Ph. D. 1975, Case Western Reserve University, Analytical Chemistry
M.S. 1972, Indian Institute of Technology, Madras, Organic Chemistry
B.S. 1970, University of Madras, Chemistry

PROFESSIONAL ORGANIZATIONS

Member, American Chemical Society.

Kendall Lindquist

Operations Manager

RELEVANT EXPERIENCE

Mr. Kendall Lindquist, Operations Manager (since 1996) and one of the owners has served as a Project Manager since 1994. He managed the Department of Army Contract for the sampling, analysis, and reporting of Polychlorinated Biphenyls in 2500 samples from an army ammunition plant. He supervises analysts and technicians in sampling procedures, sample extractions and analysis on GC and GC/MS. He is responsible for scheduling field and analytical activities and for the operation of GC and GC/MS equipment, as necessary, for the analysis of volatiles and semivolatiles by SW-846 methodologies. His past and current responsibilities include completing all analytical activities on time with adherence to holding times and due dates. He has developed and implemented numerous laboratory Standard Operating Procedures.

From 1990 to 1995 he served as a senior organic analyst with Mantech Environmental, on their EPA Region VII ESAT team. He analyzed environmental samples using EPA regional Standard Operating Procedures for volatiles, semivolatiles, pesticides/PCBs and dioxins/furans. He generated data reports in CLP-like formats and reviewed CLP-data generated by the ESAT team using regional functional guidelines. He prepared and analyzed several thousand samples for 2,3,7,8-TCDD, polychlorinated dibenzodioxins and furans (PCDDs/PCDFs) on low resolution GC/MS. He also prepared TCDD reports using CLP deliverables software.

Mr. Kendall Lindquist is proficient in Windows, WordPerfect, Excel, Lotus 123 and Microsoft Office. His knowledge of various laboratory instrument software programs (Hewlett Packard Unix/Chemstation, Hewlett Packard RTE-A, etc.) is extensive
Mr. Lindquist has attended the 40-hour OSHA health and safety training course and subsequent 8-hour refresher training courses.

EDUCATION

MBA in Management, 1993, Keller Graduate School, Kansas City, Missouri
B.S. Chemistry, 1990, Avila College, Kansas City, Missouri

Last Page of AML Laboratory QAP

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Analytical Management Laboratories, Inc.
15130 South Keeler
Olathe, Kansas 66062
Phone (913) 829-0101
Fax (913) 829-1181

31173

Chain of Custody Record / Request for Analysis

Client Contact Name:_____
Company Name:_____
Address:_____
City, State, Zip:_____
Phone #:( ) _____ Ext: _____
Fax #:( ) _____
Email:_____

Project Name:_____
Project Number:_____
Purchase Order Number:_____
Project Due Date:_____
Project Comments:_____
Sampler's Signature:_____

Analyses/Method to be Performed (Check all that apply)

Laboratory Project Number: _____

Specify method # --->

Lab ID	Sample Description	Date	Time	Matrix	Total # Containers	HCl	HNO_3	NaOH	H_2SO_4	Unpreserved	4° C		VOCs	SVOCs	Pesticides	PCBs	Herbicides	Metals (specify)	DRO	GRO	Explosives	Anions (specify)								Comments:
						Preservative List total number of bottles for each preservative type.																								Please include any information that may be useful in the analysis of the sample. Example: high concentration List analytes MS/MSD
1																														
2																														
3																														
4																														
5																														
6																														
7																														
8																														
9																														
10																														

CUSTODY							
Relinquished By:		Date/Time:		Received By:		Date/Time:	
Relinquished By:		Date/Time:		Received By:		Date/Time:	

By signing the request (chain of custody) you are ordering work from Analytical Management Laboratories, Inc. which constitutes the acceptance of the terms and conditiions on the back of this form.

Delivery Method	Custody Seals	Coolant	Cooler Temp.	Receiving Comments:
☐ Delivered in Person ☐ Courier _____	☐ Yes ☐ No ☐ Intact	☐ Ice ☐ Blue Ice	_____ °C ☐ Temp. Blank	

U.S. ARMY CORPS OF ENGINEERS

CHAIN OF CUSTODY RECORD 5542

PROJ. NO.: 57

PROJECT NAME: Forbes 57

SAMPLERS: (Signature)

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	VOCs 8260B						REMARKS
	8/3	1125		✓	57Ø5MW1D-Ø1	3	X		5542-01				Cool 4°C, pH<2.0 (HCL
	"	"		✓	57Ø5MW1D-MS	3	X		5542-01				" "
	"	"		✓	57Ø5MW1D-MSD	3	X		5542-01				" "
	"	1230		✓	57Ø5MW1S-Ø1	3	X		5542-02				" "
	"	1410		✓	57Ø5MW5S-Ø1	3	X		5542-03				" "
	"	1505		✓	57Ø5MW5D-Ø1	3	X		5542-04				" "
	"	1605		✓	57Ø5MW8D-Ø1	3	X		5542-05				" "
	—	—		✓	Trip Blank	2	X		5542-06				" "
	—	—		—	Temp Blank	1							Cool 4°C

Notes follows

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
			08/04/04 0923am		
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

ical Management Laboratories,

AML - Sample Condition Upon Receipt Report

Client ID: USACEOK

AML Work Order Number: 5342

Project ID: Former Atlas Missile Site 7

Cooler ID: _____

Delivery Method

Delivery Method: Courier

Name of Person Receiving Samples: NS

Couirer ID: Federal Express

Airbill Number: 846325565853

Custody Seals

Were Custody Seals Present? ☑

Cooler Opened By: NS

Were Custody Seals Intact? ☑

Date Opened: 6/25/04

Number of Custody Seals: 2

Coolant / Temperature

Type of Coolant Used: Ice

Temperature of Cooler: 2

Temperature Taken From: Cooler

Chain of Custody

Was Chain of Custody filled out properly? ☑

Do Chain of Custody and Sample Labels agree? ☑

Comments

Type of Packing Used? Bubble Wrap

Were all sample labels complete? ☑

Were all bottles sealed in separate plastic bags? ☑

Were correct preservatives added to the samples? ☑

Did all the bottles arrive unbroken? ☑

Were air bubbles absent in VOA samples? ☑ N/A ☐

Was a sufficient amount of sample sent for analysis? ☑

Was project manager contacted about any "out of control" issues? ☐

EDD (if applicable) Type: _____

☐ None ☐ ERPMS ☐ Excel

☐ ITEMS ☐ Access 97 ☑ Access 2000

Samples Received by: __*NS*_____

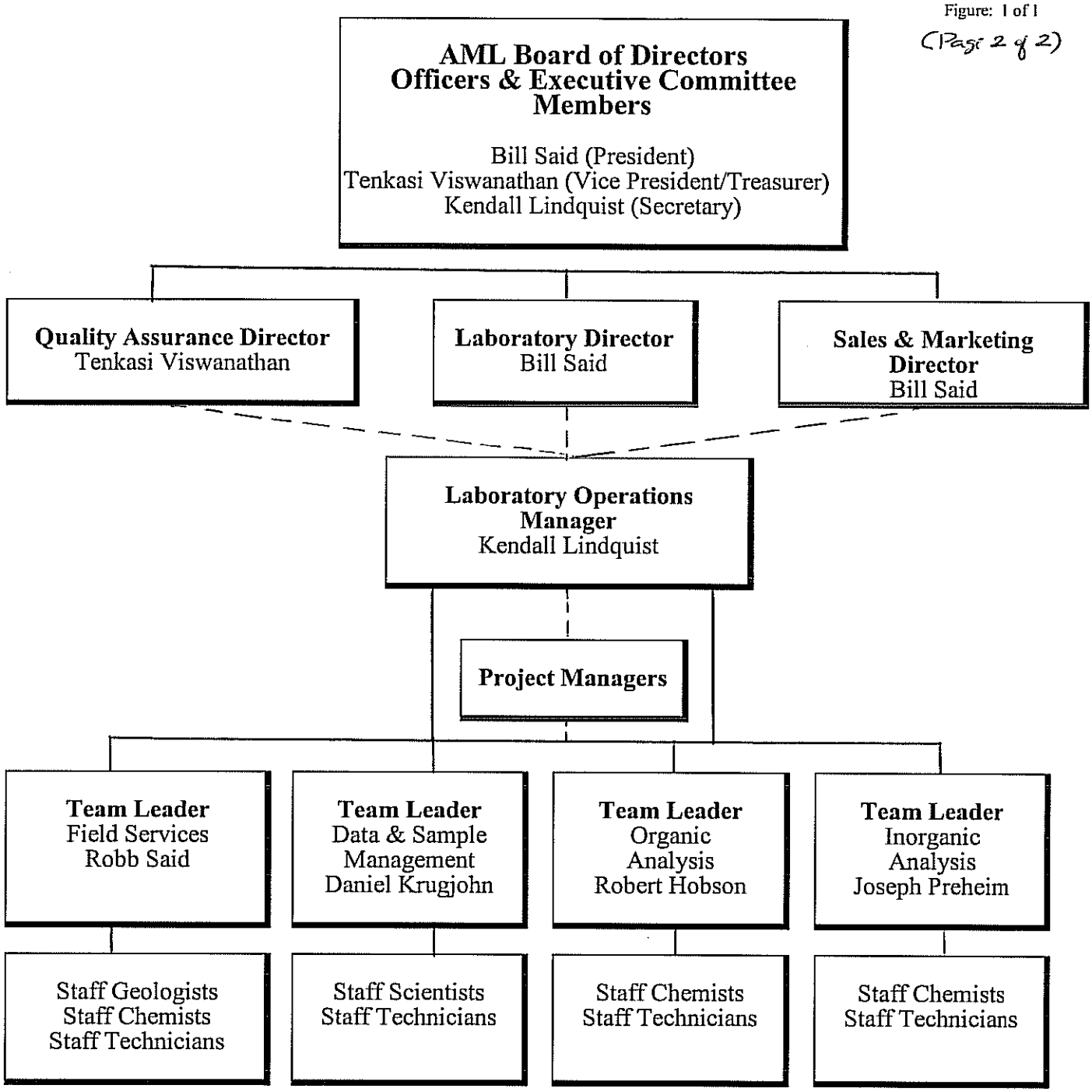
Project Manager Review: _____

Date: 6/28/04

Date: _____

Figure 4.1

Organizational Chart


AML Board of Directors
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Tenkasi Viswanathan (Vice President/Treasurer)
Kendall Lindquist (Secretary)
Quality Assurance Director
Tenkasi Viswanathan
Laboratory Director
Bill Said
Sales & Marketing Director
Bill Said
Laboratory Operations Manager
Kendall Lindquist
Project Managers
Team Leader
Field Services
Robb Said
Team Leader
Data & Sample Management
Daniel Krugjohn
Team Leader
Organic Analysis
Robert Hobson
Team Leader
Inorganic Analysis
Joseph Preheim
Staff Geologists
Staff Chemists
Staff Technicians
Staff Scientists
Staff Technicians
Staff Chemists
Staff Technicians
Staff Chemists
Staff Technicians

Figure 7.1

Preservatives, Holding Times, and Sample Containers

(Reproduced from Appendix B, USACE EM 200-1-3, February 2001)

QAP Figure 7.1

Appendix B
Preservatives, Holding Times, and Sample Containers

Table B-1
Preservatives, Holding Times, and Sample Containers for SW-846 Methods[1]

	Preservatives[3]		Holding Time		Containers[4]	
Parameter[2]	Aqueous Liquid	Solid	Aqueous Liquid	Solid	Aqueous Liquid	Solid
Volatile Organics[5] (VOCs) Including: 8260 - VOCs 8021 - Aromatic VOCs 8021 - Halogenated VOCs 8015 - Nonhalogenated VOCs 8032 - Acrylamide	No Residual Chlorine present Adjust pH to < 2 with H_2SO_4, HCl, or solid sodium bisulfate ($NaHSO_4$). Cool to 4 °C.	Low-level soil[6] Add ~5 g soil to 40-mL VOA vial pre-preserved with 1 g of $NaHSO_4$/5 mL water.	14 days	Low/High Level 14 days	3 × 40 mL glass VOA vial, PTFE septa cap	3 × 40 (or 60) mL glass VOA vial (with stir bar for low-level soil), PTFE septa cap[7]

N/A - Not Applicable.
[1] Additional standard method manuals may be applicable. Refer to individual methods employed for details.
[2] Major chemical parameter headings (i.e., Volatile Organics, Extractable Organics, Metals, etc.) are given in boldface type. When applicable, subset chemical parameters and method numbers have been added to clarify that the same preservative, container, and holding time requirements apply. When multiple chemical parameters or chemical parameter subsets are needed, the specified number of sample containers must be submitted to the laboratory for each chemical analysis requested.
[3] Reagents should be of appropriate grade or quality as identified within the analytical method.
[4] All containers must have PTFE (Teflon)-lined seals (Teflon-lined septa for volatile organic analyte (VOA) vials).
[5] VOC section is segregated based on preparatory procedures employed. Refer to SW-846 Method 5000 for details on determinative methods application to appropriate preparatory procedures.
[6] Refer to SW-846 Method 5035 for additional preservation options.
[7] Actual number of sample containers necessary for each location is based on project information. Refer to Instruction E-5 in Appendix E for additional details.

(Sheet 1 of 9)

QAP Figure 7.1
Page 3 of 10

Table B-1 (Continued)

Parameter[2]	Preservatives[3]		Holding Time		Containers[4]	
	Aqueous Liquid	Solid	Aqueous Liquid	Solid	Aqueous Liquid	Solid
VOCs (Cont.) 8033 - Acetonitrile 8315 - Carbonyl Compounds[8] Prepared by: 5030 - Purge and trap (aqueous) 5035 - Closed system purge and trap (solid)	Residual Chlorine present Collect sample in a 125-mL container, prepreserved with 4 drops of 10% sodium thiosulfate ($Na_2S_2O_3$) solution. Gently swirl to mix, and transfer to 40-mL VOA vials. Adjust pH to < 2 with H_2SO_4, HCl, or solid $NaHSO_4$. Cool to 4 °C, no headspace.	High-level soil Add ~5 g soil to 40- (or 60-) mL VOA vial prepreserved with 10 mL methanol. Concentrated waste[9] Collect sample in a 40- (or 60-) mL VOA vial with minimal headspace. Cool to 4 °C. EnCore™ Sampler (or similar) Collect 5-g samples according to manufacturer's instructions.		EnCore™ Sampler 48 hours		3 - EnCore™ Sampler (or similar)[7]
Prepared by: 5021 - Automated Headspace	N/A	Soil only Add ~2 g soil to 22-mL soil vial. Cool to 4 °C. Soil/Matrix Modifier[10] Add ~2 g soil to 22-mL soil vial prepreserved with 10 mL matrix modifier. Cool to 4 °C. Soil/Water Add ~2 g soil to 22-mL soil vial prepreserved with 10 mL water. Cool to 4 °C.	N/A	14 days	N/A	2 × 22 mL glass soil headspace vial, PTFE-lined septa with crimp or screw-top cap[7]
Prepared by: 5032 - Vacuum Distillation	Same as VOC - purge and trap.	Cool to 4 °C. No headspace.	14 days	14 days	2 × 40 mL glass vial, PTFE septa cap	2 × 125 - mL CWM[11]

[8] Samples must be derivatized and extracted within 3 days of collection and analyzed within 3 days of extraction.
[9] Refer to SW-846 Method 5035 for additional preservation options for oily waste.
[10] Refer to SW-846 Method 5021 for details on the matrix modifying solution needed.
[11] CWM - Clear wide-mouth glass jar(s) with Teflon-lined lid(s).

(Sheet 2 of 9)

QAP Figure 7.1 Page 4 of 10

Table B-1 (Continued)

Parameter[2]	Preservatives[3]		Holding Time		Containers[4]	
	Aqueous Liquid	Solid	Aqueous Liquid	Solid	Aqueous Liquid	Solid
Nonpurgeable Water-Soluble VOCs Prepared by: 5031 - Azeotropic Distillation	Same as VOC - purge and trap.	Cool to 4 °C, No headspace.	14 days	14 days	2 × 40 mL glass vial, PTFE septa cap	2 × 125 - mL CWM[11]
VOCs Prepared by: 3585 - Solvent Dilution	N/A	Oily waste Cool to 4 °C.	N/A	14 days	N/A	125-mL CWM[11]
VOCs Including: 8031 - Acrylonitrile 8316 - Acrolein, Acrylamide, Acrylonitrile	Adjust pH to 4-5 with H_2SO_4, HCl, or solid $NaHSO_4$. (Cool to 4 °C.	N/A	14 days	N/A	2 × 40 mL glass vial, PTFE septa cap	N/A
Extractable Organics Including: 8270 - Semivolatile Organics 8041 - Phenols[13] 8061 - Phthalate Esters 8070 - Nitrosamines[14] 8081 - Organochlorine Pesticides[14] 8082 - Polychlorinated Biphenyls[14] 8091 - Nitroaromatics / Cyclic Ketones[14] 8100 - PAHs[14] 8111 - Haloethers[14] 8121 - Chlorinated Hydrocarbons 8151 - Chlorinated Herbicides 8310 - PAHs 8321 - Nonvolatile Organics 8325 - Nonvolatile Organics[12] 8330 - Nitroaromatics and Nitramines (explosives)[14] 8331 - Tetrazene [14] 8332 - Nitroglycerine	No Residual Chlorine present Cool to 4 °C. Residual Chlorine present Add 1 mL 10% sodium thiosulfate ($Na_2S_2O_3$) solution per liter water. Cool to 4 °C.	Cool to 4 °C.	7d/40d[15]	14d/40d[16]	2 × 1 L AG[17] per chemical parameter	250 mL CWM[11,18,19]

[11] CWM - Clear wide-mouth glass jar(s) with Teflon-lined lid(s).
[12] Extracts must be stored at -10°C and in the dark until analysis.
[13] Extracts should be methylated within 48 hours of extraction, and analyzed immediately thereafter.
[14] Extracts must be stored at 4°C and in the dark until analysis.
[15] 7 days until extraction/analyzed within 40 days after extraction.
[16] 14 days until extraction/analyzed within 40 days after extraction.
[17] AG - Amber Glass bottle(s) with Teflon-lined cap(s).
[18] The subset chemical parameters under Extractable Organics may be collected in the same container for soils.
[19] High-concentration waste samples do not need cooling.

(Sheet 3 of 9)

QAP Figure 7.1
Page 5 of 10

Table B-1 (Continued)

Parameter[2]	Preservatives[3]		Holding Time		Containers[4]	
	Aqueous Liquid	Solid	Aqueous Liquid	Solid	Aqueous Liquid	Solid
Extractable Organics 8141 - Organophosphate Pesticides	Adjust to 5 < pH < 9 with H_2SO_4 or NaOH. Cool to 4 °C.	Cool to 4 °C.	7d/40d[15]	14d/40d[16]	2 × 1 L AG[17]	250 mL CWM[11]
Extractable Organics 8318 - N-Methylcarbamates	Adjust pH to 4-5 with 0.1 N chloroacetic acid. Cool to 4 °C. Store in dark.	Cool to 4 °C. Store in dark.	7d/40d[15]	14d/40d[16]	2 × 1 L AG[17]	250 mL CWM[11]
Extractable Organics 8280 - Dioxins / Furans 8290 - Dioxins / Furans	<u>No Residual Chlorine present</u> If sample pH is greater than 9, adjust to pH 7-9 with sulfuric acid (H_2SO_4) Cool to 4 °C. Store in dark. <u>Residual Chlorine present</u> Add 80 mg sodium thiosulfate ($Na_2S_2O_3$) per liter water. If sample pH is greater than 9, adjust to pH 7-9 with sulfuric acid (H_2SO_4). Cool to 4 °C. Store in dark.	Cool to 4 °C. Store in dark.	30d/45d[20]	30d/45d[20]	2 × 1 L AG[17]	250 mL CWM[11]
Extractable Organics 8131 - Analine / selected derivatives	<u>No Residual Chlorine present</u> Adjust pH to 6-8 with H_2SO_4 or NaOH. Cool to 4 °C. <u>Residual Chlorine present</u> Add 35 mg sodium thiosulfate ($Na_2S_2O_3$) per ppm chlorine per liter water. Adjust pH to 6-8 with H_2SO_4 or NaOH. Cool to 4 °C.	Cool to 4 °C.	7d/40d[15]	14d/40d[16]	2 × 1 L AG[17]	250 mL CWM[11]

[11] CWM - Clear wide-mouth glass jar(s) with Teflon-lined lid(s).
[15] 7 days until extraction/analyzed within 40 days after extraction.
[16] 14 days until extraction/analyzed within 40 days after extraction.
[17] AG - Amber Glass bottle(s) with Teflon-lined cap(s).
[20] 30 days until extraction/analyzed within 45 days after extraction.

(Sheet 4 of 9)

QAP Figure 7.1 Page 6 of 10

Table B-1 (Continued)

Parameter[2]	Preservatives[3]		Holding Time		Containers[4]	
	Aqueous Liquid	Solid	Aqueous Liquid	Solid	Aqueous Liquid	Solid
Metals (Except Chromium (VI) & Mercury)	HNO_3 to pH < 2.	Cool to 4 °C.	6 months	6 months	1 L HDPE[21]	250 mL CWM[11,22]
Mercury	HNO_3 to pH < 2.	Cool to 4 °C.	28 days	28 days	250 mL HDPE[21] or glass	250 mL CWM[11]
Chromium (VI)	Cool to 4 °C.	Cool to 4 °C.	24 hours	24 hours	250 mL HDPE[21]	250 mL CWM[11]
Miscellaneous						
Acidity	Cool to 4 °C.	N/A	48 hours	N/A	250 mL HDPE[21]	N/A
Alkalinity	Cool to 4 °C.	N/A	48 hours	N/A	250 mL HDPE[21]	N/A
Ammonia	Cool to 4 °C, H_2SO_4 to pH < 2.	N/A	14 days	N/A	1 L HDPE[21]	N/A
Biochemical Oxygen Demand (BOD)	Cool to 4 °C, H_2SO_4 to pH < 2.	N/A	48 hours	N/A	2 L HDPE[21]	N/A
Biochemical Oxygen Demand (Carbonaceous)	Cool to 4 °C.	N/A	48 hours	N/A	2 L HDPE[21]	N/A
Bromide	Cool to 4 °C.	N/A	28 days	N/A	250 mL HDPE[21]	N/A
Chemical Oxygen Demand	Cool to 4 °C, H_2SO_4 to pH < 2.	N/A	28 days	N/A	125 mL HDPE[21]	N/A
Chloride	None Required.	None Required.	28 days	28 days	125 mL HDPE[21]	125 mL CWM[11]
Chloride (Total Residual)	None Required.	N/A	ASAP	N/A	500 mL HDPE[21]	N/A
Coliform, Fecal & Total	Cool to 4 °C (add 0.1 mL of 10% $Na_2S_2O_3$ if residual Cl present).	Cool to 4 °C.	6 hours	6 hours	120 mL HDPE[21]	125 mL CWM[11]
Color	Cool to 4 °C.	N/A	48 hours	N/A	125 mL HDPE[21]	N/A
Common Ions	Cool to 4 °C.	Cool to 4 °C.	28 days	28 days	1 L glass	250 mL CWM[11]

[11] CWM - Clear wide-mouth glass jar(s) with Teflon-lined lid(s).
[21] HDPE - High-Density Polyethylene bottles.
[22] Metals and cyanide may be collected in the same container for soils.

(Sheet 5 of 9)

QAP Figure 7.1 Page 7 of 10

Table B-1 (Continued)

Parameter[2]	Preservatives[3]		Holding Time		Containers[4]	
	Aqueous Liquid	Solid	Aqueous Liquid	Solid	Aqueous Liquid	Solid
Miscellaneous (Cont.)						
Cyanide (Total & Amenable)	Cool to 4 °C and adjust pH > 12 with 50% NaOH If Oxidizing Agents are present: Cool to 4 °C. Add 5 mL 0.1 N $NaAsO_2$ per liter water or 0.06 g ascorbic acid per liter water. Adjust pH > 12 with 50% NaOH.	Cool to 4 °C.	14 days	14 days	1 L HDPE[21]	250 mL CWM[11, 22]
Dissolved Oxygen, Winkler Method	Fix on Site/Store in dark.	N/A	8 hours	N/A	300 mL BOD bottle.	N/A
Fecal Streptococci	Cool to 4 °C (0.008% $Na_2S_2O_3$ if residual Cl present).	N/A	6 hours	N/A	250 mL HDPE[21]	N/A
Fluoride	Cool to 4 °C.	N/A	28 days	N/A	500 mL HDPE[21]	N/A
Hardness	HNO_3 or H_2SO_4 to pH < 2.	N/A	6 months	N/A	250 mL HDPE[21]	N/A
Hydrogen Ion (pH)	None Required.	Cool to 4 °C.	24 hours	ASAP	60 mL HDPE[21]	125 mL CWM[11]
Kjeldahl and Organic Nitrogen	Cool to 4 °C, H_2SO_4 to pH < 2.	N/A	28 days	N/A	1 L HDPE[21]	N/A
Nitrate	Cool to 4 °C.	Cool to 4 °C.	48 hours	48 hours	250 mL HDPE[21]	250 mL CWM[11]
Nitrate-Nitrite	Cool to 4 °C, H_2SO_4 to pH < 2.	Cool to 4 °C.	28 days	28 days	250 mL HDPE[21]	250 mL CWM[11]
Nitrite	Cool to 4 °C.	N/A	48 hours	N/A	125 mL HDPE[21]	N/A
Oil & Grease	Cool to 4 °C and add 5 mL 1:1 (HCl).	Cool to 4 °C.	28 days	28 days	2 - 1 l glass	250 mL CWM[11]

[11] CWM - Clear wide-mouth glass jar(s) with Teflon-lined lid(s).
[21] HDPE - High-Density Polyethylene bottles.
[22] Metals and cyanide may be collected in the same container for soils.

(Sheet 6 of 9)

QAP Figure 7.1 Page 8 of 10

Table B-1 (Continued)

Parameter[2]	Preservatives[3]		Holding Time		Containers[4]	
	Aqueous Liquid	Solid	Aqueous Liquid	Solid	Aqueous Liquid	Solid
Miscellaneous (Cont.) Organic Carbon, Total (TOC)	Adjust pH to < 2 with H_2SO_4, HC1, or solid sodium bisulfate ($NaHSO_4$). Cool to 4 °C. Store in dark.	Cool to 4 °C.	28 days	28 days	125 mL HDPE[21]	125 mL CWM[11]
Orthophosphate	Filter immediately. Cool to 4 °C.	N/A	48 hours	N/A	125 mL HDPE[21]	N/A
Phenolics	Cool to 4 °C and adjust to pH < 4 with H_2SO_4.	Cool to 4 °C.	28 days	28 days	1 L BR[23]	250 mL CWM[11]
Phosphorus (Elemental)	Cool to 4 °C.	N/A	48 hours	N/A	1 L BR[23]	N/A
Phosphorus (Total)	Cool to 4 °C, H_2SO_4 to pH < 2.	N/A	28 days	N/A	125 mL HDPE[21]	N/A
Radiological Test, Gross Alpha	HNO_3 to pH < 2.	Cool to 4 °C.	6 months	6 months	2 L HDPE[21]	250 mL HDPE[21]
Radiological Test, Gross Beta	HNO_3 to pH < 2.	Cool to 4 °C.	6 months	6 months	2 L HDPE[21]	250 mL HDPE[21]
Radiological Radium (Total)	HNO_3 to pH < 2.	Cool to 4 °C.	6 months	6 months	2 L HDPE[21]	250 mL HDPE[21]
Residue (Filterable)	Cool to 4 °C.	N/A	7 days	N/A	250 mL HDPE[21]	N/A
Residue (Nonfilterable)	Cool to 4 °C.	N/A	7 days	N/A	250 mL HDPE[21]	N/A
Residue (Settleable)	Cool to 4 °C.	N/A	48 hours	N/A	Imhoff Cone	N/A
Residue (Total)	Cool to 4 °C.	N/A	7 days	N/A	250 mL HDPE[21]	N/A
Residue (Volatile)	Cool to 4 °C.	N/A	7 days	N/A	250 mL HDPE[21]	N/A
Silica	Cool to 4 °C.	N/A	28 days	N/A	125 mL HDPE[21]	N/A
Specific Conductance	Cool to 4 °C.	N/A	28 days	N/A	250 mL HDPE[21]	N/A
Sulfate	Cool to 4 °C.	Cool to 4 °C.	28 days	28 days	125 mL HDPE[21]	125 mL CWM[11]

[11] CWM - Clear wide-mouth glass jar(s) with Teflon-lined lid(s).
[21] HDPE - High-Density Polyethylene bottles.
[22] Metals and cyanide may be collected in the same container for soils.
[23] BR - Boston Round.

(Sheet 7 of 9)

App Figure 7.1
Page 9 of 10

Table B-1 (Continued)

Parameter[2]	Preservatives[3]		Holding Time		Containers[4]	
	Aqueous Liquid	Solid	Aqueous Liquid	Solid	Aqueous Liquid	Solid
Miscellaneous (Cont.)						
Sulfide	Cool to 4 °C, add 4 drops zinc acetate, and NaOH to pH > 9.	Add 2 N zinc acetate until moistened, and cool to 4 °C.	7 days	7 days	1 L HDPE[21]	250 mL CWM[11]
Sulfite	Cool to 4 °C.	N/A	ASAP	N/A	125 mL HDPE[21]	N/A
Surfactant	Cool to 4 °C.	N/A	48 hours	N/A	500 mL HDPE[21]	N/A
TCLP Extractable Fraction	Cool to 4 °C.	Cool to 4 °C.	14 days / NA / 14 days[24]	14 days / N/A / 14 days[24]	3 - 1 L AG[17]	500 mL CWM[11]
TCLP Volatile Fraction	Cool to 4 °C. No headspace.	Cool to 4 °C, No headspace.	14 days/7 days/ 40 days[24]	14 days/7 days/ 40 days[24]	500 mL glass PTFE-lined septa	125 mL glass with PTFE-lined septum or 25 g EnCore™ sampler
TCLP Inorganic Fraction (Mercury)	Cool to 4 °C.	Cool to 4 °C.	28 days / N/A / 28 days[24]	28 days / N/A / 28 days[24]	1 L HDPE[21]	500 mL CWM[11]
TCLP Inorganic Fraction (all other metals)	Cool to 4 °C.	Cool to 4 °C.	180 days / N/A / 180 days[24]	180 days / N/A/ 180 days[24]	1 L HDPE[21]	500 mL CWM[11]
Temperature	None required.	N/A	ASAP	N/A		
Total Organic Halogens (TOX)	Cool to 4 °C, H_2SO_4 to pH < 2.	Cool to 4 °C.	28 days	28 days	16 oz BR[23]	125 mL CWM[11]
Toxic Recoverable Petroleum Hydrocarbons	Cool to 4 °C, H_2SO_4 to pH < 2.	Cool to 4 °C.	28 days	28 days	2 - 1 L glass	250 mL CWM[11]
Turbidity	Cool to 4 °C.	N/A	48 hours	N/A	250 mL HDPE[21]	N/A

[11] CWM - Clear wide-mouth glass jar(s) with Teflon-lined lid(s).
[17] AG - Amber Glass bottle(s) with Teflon-lined cap(s).
[21] HDPE - High-Density Polyethylene bottles.
[23] BR - Boston Round.
[24] Holding times shown: from collection to toxicity characteristic leaching procedure (TCLP) extraction/from TCLP extraction to preparative procedure/from preparative procedure to analysis.

(Sheet 8 of 9)

QAP Figure 7.1 Page 10 of 10

QAP Figure 7.2
(Page 1 of 5)

Figure 7.2

Chain of Custody/Log-in Record

AML COC Record/Request for Analysis Form

USACE Chain of Custody Record

Shaw Group Chain of Custody Record

AML
Analytical Management Laboratories, Inc.
15130 B South Keeler
Olathe, Kansas 66062
Phone (913) 829-0101
Fax (913) 829-1181

24189

Chain of Custody Record / Request for Analysis

Client Contact Name: Catherine Drumheller
Company Name: ECC
Address: 1746 Cole Blvd Bld 21 Suite 350
City, State, Zip: Lakewood CO 80401
Phone #: (303) 298-7607
Fax #: (303) 298-7532

Project Name: ENOP Mead NE
Project Number: 5430-008
Purchase Order Number:
Project Due Date: 30 Days
Project Comments:
Sampler's Signature:

Analyses/Method to be Performed (Check all that apply)

Laboratory Project Number: 6391

Method # --->

Lab ID	Sample Description	Date	Time	Matrix	Total # Containers	HCl	HNO_3	NaOH	H_2SO_4	Unpreserved	4°C	TPH Diesel	TPH Gasoline	BTEX	MTBE	Volatiles (VOCs) 8260B	BNAs (SVOCs)	Pesticides/PCBs	PCBs	RCRA8 Metals	Lead	Flash Point	Paint Filter	pH	Explosives 8330	TSS 160.2	Metals 6010B	Comments:
6391-01	5430-0204 EW2-002	2-3-04	9:10	GW	7	3	1				3					X									X	X	X	
6391-02	5430-0204 EW3-003		9:30		7	3	1				3					X									X	X	X	
6391-03	5430-0204 EW4-004		9:23		7	3	1				3					X									X	X	X	
6391-04	5430-0204 EW5-005		11:40		7	3	1				3					X									X	X	X	
6391-05	5430-0204 EW6-006		11:08		7	3	1				3					X									X	X	X	
6391-06	5430-0204 EW7-007		11:00		7	3	1				3					X									X	X	X	
6391-07	5430-0204 EW8-008		10:05		7	3	1				3					X									X	X	X	
6391-08	5430-0204-2 EW8-009		10:05		7	3	1				3					X									X	X	X	
6391-07	5430-0204-EW8-MS		10:05		6	3	1				2					X									X		X	
6391-07	5430-0204 EW8-MSD		10:05		6	3	1				2					X									X		X	

Please include any information that may be useful in the analysis of the sample.

Example: high concentration

CUSTODY						
Relinquished By:		Date/Time:	2-3-04 1400	Received By:	Fed Ex Air Bill 8396 6431 5842	Date/Time: 2-3-04 1400
Relinquished By:		Date/Time:		Received By:		Date/Time: 02-04-03 09:00am

By signing the request (chain of custody) you are ordering work from Analytical Management Laboratories, Inc. which constitutes the acceptance of the terms and conditions on the back of this form.

Delivery Method	Custody Seals	Coolant	Cooler Temp.	Receiving Comments:
☐ Delivered in Person ☐ Courier FedEx ☐ Airbill # 8396 6431 5842	☑ Yes ☐ No ☑ Intact ☐ Broken	☑ Ice ☐ Blue Ice ☐ None	°C ☑ Temp. Blank ☐ Scan	→ Cooler #1: 2.7°C Cooler #3: 3.3°C Cooler #5: 2.6°C Cooler #2: 2.9°C Cooler #4: 2.3°C

QA 17 Sup 7.2 - Page 2 of 5

AML
Analytical Management Laboratories, Inc.
15130 B South Keeler
Olathe, Kansas 66062
Phone (913) 829-0101
Fax (913) 829-1181

24188

Chain of Custody Record / Request for Analysis

Client Contact Name: Catherine Drumheller
Company Name: ECC
Address: 1746 Cole Blvd Bld 21 Suite 350
City, State, Zip: Lakewood Co 80401
Phone #: (303) 298-7602
Fax #: (303) 298-7537

Project Name: FNOP Mead NE
Project Number: 5430.008
Purchase Order Number:
Project Due Date: 30 days
Project Comments:
Sampler's Signature:

Analyses/Method to be Performed (Check all that apply)

Laboratory Project Number: 6391

Method # --->

Lab ID	Sample Description	Date	Time	Matrix	Total # Containers	HCl	HNO_3	NaOH	H_2SO_4	Unpreserved	4°C	TPH Diesel	TPH Gasoline	BTEX	MTBE	Volatiles (VOCs) 8260B	BNAs (SVOCs)	Pesticides/PCBs	PCBs	RCRA8 Metals	Lead	Flash Point	Paint Filter	pH	Explosives 8330	TSS 160.2	Metals 6010B	Comments:
6391-09	5430.0304.EW9.011	2.3.04	10:52	GW	2	3	1				3					X									X	X	X	
6391-10	5430 0304 EW10.012	2.3.04	10:45	GW	2	3	1				3					X									X	X	X	
6391-11	GCW1-INF.022004	2.3.04	13:45	GW	3	3										X												
6391-12	GCW1.INF.022004	2.3.04	13:45	GW	3	3										X												
6391-13	GCW1.EFF.022004	2.3.04	13:50	GW	3	3										X												
6391-14	GCW2.INF.022004	2.3.04	11:55	GW	2						2														X			
6391-15	GCW2.INF.022004	2.3.04	11:55	GW	2						2														X			
6391-16	GCW2.EFF.022004	2.3.04	12:05	GW	2						2														X			
6391-17	Trip Blank				2	2										X												
10																												

Please include any information that may be useful in the analysis of the sample.
Example: high concentration

CUSTODY					
Relinquished By:	[signature]	Date/Time: 2-3-04 1400	Received By:	Fed Ex A:1 B:11 8391 6431 5480	Date/Time: 2-3-04 1400
Relinquished By:		Date/Time:	Received By:	[signature]	Date/Time: 02-04-03 0900am

By signing the request (chain of custody) you are ordering work from Analytical Management Laboratories, Inc. which constitutes the acceptance of the terms and conditions on the back of this form.

Delivery Method	Custody Seals	Coolant	Cooler Temp.	Receiving Comments:
☐ Delivered in Person ☒ Courier Fedex ☐ Airbill #: See Sample Rec Form	☐ Yes ☐ No ☐ Intact ☐ Broken	☐ Ice ☐ Blue Ice ☐ None	°C ☒ Temp. Blank ☐ Cooler	Cooler #1: 2.7°C Cooler #3: 3.3°C. Cooler #5: 2.6°C Cooler #2: 2.2°C Cooler #4: 3.3

QAP Figure 7.2 - Page 3 of 5

U.S. ARMY CORPS OF ENGINEERS 4228

CHAIN OF CUSTODY RECORD

PROJ. NO.

PROJECT NAME: Bay Creek

SAMPLERS: (Signature) [signature]

STA. NO.	DATE 2003	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	VOC - 8260B	GRO - 8015B	TOC - 415.1/9060	Cu, Pb, Zn - 6020A			REMARKS		
	12/17	0925		X	BC-EW-37	3	X						40 ml vials	water	HCl
	↓	0920		X	BC-EW-19	3	X								
	—	—		X	BC-TB-121703	2	X								
	12/17	1155		X	BC-EW-8	3	X								
		1205		X	BC-EW-34	3	X								
		1335		X	BC-EW-10	3	X								
		1300		X	BC-EW-11	3	X								
		1035		X	BC-EW-9	3	X								
				X		3		X							
				X		2			X						H_2SO_4
		1055		X	BC-EW-35	3	X								HCl
				X		3		X							
				X		2			X						H_2SO_4
						1				X			500 ml poly		HNO_3

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
[signature]	12/17/03 1635	[signature] 12-18-03 12pm			
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

QAP Figure 7.2 - Page 4 of 5

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Shaw™
Shaw E & I, Inc.

ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD

Reference Document No: 010-020904-AML

Project Number: 843882
Project Name: SFAAP
Sample Coordinator: Laura Price
Turnaround Time: 14 DAY

Samples Shipment Date: 10 FEB 2004
Lab Destination: Analytical Management Laboratories
Lab Contact: Sample Receiving
Project Contact: Laura Price
Carrier/Waybill No.: COURIER/

Bill To: Angela Malone
4400 College Blvd., Suite 350
Overland Park KS 66211

Report To: Laura Price
36425 W. 103rd St.
Desoto KS 66018

Special Instructions: Cost code#08120160

Possible Hazard Indentification: Radiological ☐
Non-hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown ☒

Sample Disposal:
Return to Client ☐ Disposal by Lab ☒ Archive (mos.)

1. Relinquished By (Signature/Affiliation)	Date: 2/10/04 Time: 14:10	1. Received By (Signature/Affiliation)	Date: 2-10-04 Time: 1411
2. Relinquished By (Signature/Affiliation)	Date: Time:	2. Received By (Signature/Affiliation)	Date: Time:
3. Relinquished By (Signature/Affiliation)	Date: Time:	3. Received By (Signature/Affiliation)	Date: Time:

Comments: PO#14718; Modified Level 3 Deliverables; EDD in ITEMS Format

	Sample No	Sample Name	Sample Date	Sample Time	Container	Ctr Qty	Preservative	Requested Testing Program	Fil	CID	Condition On Receipt
413-01	010SH00062	010SO062-SS-010SH00062-REG	09 FEB 2004	11:00	9 oz CWMG	1	None except cool to 4 C	Lead by Method 6020, Nitrocellulose by USATHAMA LF03, Nitroglycerine by 8330/8332, Phthalates by SW846 8270C	N		
413-01	010SH00062	010SO062-SS-010SH00062-REG	09 FEB 2004	11:00	9 oz CWMG	1	None except cool to 4 C	Diesel range organics by OA-2, PAH	N		
413-01	010SH00062	010SO062-SS-010SH00062-REG	09 FEB 2004	11:00	5 g EasyDraw	3	None except cool to 4 C	Gasoline range organics by OA-1	N		
413-02	010SH00063	010SO063-SS-010SH00063-REG	09 FEB 2004	10:45	9 oz CWMG	1	None except cool to 4 C	Lead by Method 6020, Nitrocellulose by USATHAMA LF03, Nitroglycerine by 8330/8332	N		
413-02	010SH00063	010SO063-SS-010SH00063-REG	09 FEB 2004	10:45	9 oz CWMG	1	None except cool to 4 C	Diesel range organics by OA-2, PAH, Phthalates by SW846 8270C	N		

QAP Figure 7.2 Page 5 of 5

Figure 7.3

Sample Labels and Custody Seals

CUSTODY SEAL

DATE _____

SIGNATURE _____

QEC
Quality Environmental Containers
800-255-3950 • 304-255-3900

QEC
P.O. Box 1160
Beaver, WV 25813
800-255-3950 • 304-255-3900
Quality Environmental Containers

PROJECT NAME

SAMPLE ID	SAMPLE DATE	SAMPLE TIME
SAMPLED BY	PRESERVATIVE	
ANALYSIS REQUESTED		___ GRAB ___ COMPOSITE

(4-oz Jars & 1-L Amber)
ALL except VOA vials

QEC
P.O. Box 1160
Beaver, WV 25813
800-255-3950 • 304-255-3900
Quality Environmental Containers

PROJECT NAME

SAMPLE ID	SAMPLE DATE
SAMPLED BY	SAMPLE TIME
PRESERVATIVE	___ GRAB ___ COMPOSITE
ANALYSIS REQUESTED	

(VOA vials)

QAP Figure 7.4
(Page 1 of 4)

Figure 7.4

AML Sample Condition Upon Receipt Report

ical Management Laboratories,

AML - Sample Condition Upon Receipt Report

Client ID: ECC

AML Work Order Number: 4391

Project ID: FNOP Mead NE

Cooler ID: 3

Delivery Method

Delivery Method: Courier

Name of Person Receiving Samples: NS

Couirer ID: Federal Express

Airbill Number: 792000033609

Custody Seals

Were Custody Seals Present? ☑

Cooler Opened By: NS

Were Custody Seals Intact? ☑

Date Opened: 2/4/04

Number of Custody Seals: 1

Coolant / Temperature

Type of Coolant Used: Ice

Temperature of Cooler: 3.3

Temperature Taken From: Temperature Blank

Chain of Custody

Was Chain of Custody filled out properly? ☑

Do Chain of Custody and Sample Labels agree? ☑

Comments

Type of Packing Used? Bubble Wrap

Were all sample labels complete? ☑

Were all bottles sealed in separate plastic bags? ☐

Were correct preservatives added to the samples? ☑

Did all the bottles arrive unbroken? ☑

Were air bubbles absent in VOA samples? ☐ N/A ☑

Was a sufficient amount of sample sent for analysis? ☑

Was project manager contacted about any "out of control" issues? ☐

EDD (if applicable) Type: _____

☐ None ☐ ERPMS ☐ Excel

☐ ITEMS ☑ Access 97 ☐ Access 2000

Samples Received by: *NS*

Project Manager Review: _____

Date: 2/4/04

Date: _____

Align bottom of Airbill Pouch here.

Customer: Seal only when using a FedEx Ship or interNetShip document.

10

200

FedEx Express *USA Airbill*

FedEx Tracking Number 8391 6431 5842

MUR22

Form ID No. 0215

1 From *Please print and press hard.*

Date 2-3-04 Sender's FedEx Account Number 2512-8517-9

Sender's Name Jim Thares Phone (402) 944-2964

Company ENVIRONMENTAL CHEMICAL CORP

Address 905 COUNTY RD # 6

City ASHLAND State NE ZIP 68003

2 Your Internal Billing Reference First 24 characters will appear on invoice. ~~5303003~~ 5430.06.5

3 To

Recipient's Name Receiving Phone ( )

Company AMI

Address 15130 B South Keeler

To "HOLD" at FedEx location, print FedEx address. We cannot deliver to P.O. boxes

Address Dept

City Olathe State KS ZIP 66062

4a Express Package Service *Packages up to 150 lbs.* Delivery commitment may be later in some areas.

- [x] FedEx Priority Overnight — Next business morning
- [ ] FedEx Standard Overnight — Next business afternoon
- [ ] FedEx First Overnight — Earliest next business morning delivery to select locations
- [ ] FedEx 2Day — Second business day
- [ ] FedEx Express Saver — Third business day

FedEx Envelope rate not available. Minimum charge: One-pound rate

4b Express Freight Service *150 lbs. in areas.*

emp# 49591 03FEB04 PRIORITY OVERNIGHT WED

Deliver By: 04FEB04 A2

FedEx

Master 8391 6431 5842 FORM 0215

002/005 MPS# 7920 0003 3609 FORM 0681

MCI

66062 -KS -US 76 IXDA

NO POUCH NEEDED.

See back for peel and stick application instructions.

PULL AND RETAIN THIS COPY BEFORE AFFIXING TO THE PACKAGE.

Try online shipping at fedex.com

By using this Airbill you agree to the service conditions on the back of this Airbill and in our current Service Guide, including terms that limit our liability.

Questions? Visit our Web site at fedex.com or call 1.800.Go.FedEx® 800.463.3339.

0237773164

...unless you declare a higher value. See back for details.

FedEx Use Only

5 Release Signature *Sign to authorize delivery without obtaining signature.*

By signing you authorize us to deliver this shipment without obtaining a signature and agree to indemnify and hold us harmless from any resulting claims.

447

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QAP Figure 7-4
Page 3 of 4

10

200

FedEx Express *USA Airbill*

FedEx Tracking Number 8391 6431 5842

Date 5-7-07

NO POUCH NEEDED.
See back for peel and stick application instructions.

RECIPIENT: PEEL HERE

1 From This portion can be removed for Recipient's records.

Date 5-7-07 FedEx Tracking Number 839164315842

Sender's Name [illegible] Phone [illegible]

Company ENVIRONMENTAL [illegible]

Address 903 COUNTY RD # 6

Dept./Floor/Suite/Room

City ASHLAND State [illegible] ZIP [illegible]

2 Your Internal Billing Reference [illegible] 5430.008

3 To

Recipient's Name [illegible] Phone

Company AMD

Address 1512 B S. [illegible]

To "HOLD" at FedEx location, print FedEx address. We cannot deliver to P.O. boxes or P.O. ZIP codes.

Address

Dept./Floor/Suite/Room

City [illegible] State [illegible] ZIP [illegible]

8391 6431 5842

0237703124

Tracking No. 0215 Recipient's Copy

4a Express Package Service *Packages up to 150 lbs.* Delivery commitment may be later in some areas.

- [x] FedEx Priority Overnight — Next business morning
- [ ] FedEx Standard Overnight — Next business afternoon
- [ ] FedEx First Overnight — Earliest next business morning delivery to select locations
- [ ] FedEx 2Day — Second business day
- [ ] FedEx Express Saver — Third business day

FedEx Envelope rate not available. Minimum charge: One-pound rate

4b Express Freight Service *Packages over 150 lbs.* Delivery commitment may be later in some areas.

- [ ] FedEx 1Day Freight* — Next business day
- [ ] FedEx 2Day Freight — Second business day
- [ ] FedEx 3Day Freight — Third business day

* Call for Confirmation:

5 Packaging * Declared value limit $500

- [ ] FedEx Envelope*
- [ ] FedEx Pak* — Includes FedEx Small Pak, FedEx Large Pak, and FedEx Sturdy Pak
- [x] Other

6 Special Handling Include FedEx address in Section 3.

- [ ] SATURDAY Delivery — Available only for FedEx Priority Overnight and FedEx 2Day to select ZIP codes
- [ ] HOLD Weekday at FedEx Location — Not available for FedEx First Overnight
- [ ] HOLD Saturday at FedEx Location — Available only for FedEx Priority Overnight and FedEx 2Day to select locations

Does this shipment contain dangerous goods? One box must be checked.

- [x] No
- [ ] Yes — As per attached Shipper's Declaration
- [ ] Yes — Shipper's Declaration not required
- [ ] Dry Ice — Dry Ice, 9, UN 1845 ____ x ____ kg

Dangerous Goods (including Dry Ice) cannot be shipped in FedEx packaging.

- [ ] Cargo Aircraft Only

7 Payment *Bill to:* Enter FedEx Acct. No. or Credit Card No. below.

- [x] Sender — Acct. No. in Section 1 will be billed.
- [ ] Recipient
- [ ] Third Party
- [ ] Credit Card
- [ ] Cash/Check
- [ ] Obtain Recip. Acct. No.

Total Packages 5 Total Weight 210 lb

Total Charges

Credit Card Auth.

†Our liability is limited to $100 unless you declare a higher value. See the *FedEx Service Guide* for details.

8 Release Signature *Sign to authorize delivery without obtaining signature.*

By signing you authorize us to deliver this shipment without obtaining a signature and agree to indemnify and hold us harmless from any resulting claims.

Questions? Visit our Web site at fedex.com or call 1.800.Go.FedEx® 800.463.3339.

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Figure 7.5

AML LIMS Sample Status and Receipt Report

Analytical Management Laboratories - Sample Status and Receipt Report

AML Project Number	4391	**Client AML ID**	ECC
AML Profile Number	218	**Client Project ID**	FNOP Mead NE

AML Sample	*Matrix*	Client Sample ID	Date Collected	Projected Due Date	Procedure	Comments
439101	W	5430-0204-EW2-002	02/03/2004 9:10	03/04/2004 9:10	8330Prep	
439101	W	5430-0204-EW2-002	02/03/2004 9:10	03/04/2004 9:10	8330W	
439101	W	5430-0204-EW2-002	02/03/2004 9:10	03/04/2004 9:10	160.2WTSS	
439101	W	5430-0204-EW2-002	02/03/2004 9:10	03/04/2004 9:10	3010A	Mn Fe
439101	W	5430-0204-EW2-002	02/03/2004 9:10	03/04/2004 9:10	6010	Mn Fe
439101	W	5430-0204-EW2-002	02/03/2004 9:10	03/04/2004 9:10	8260W	
439102	W	5430-0204-EW3-003	02/03/2004 9:30	03/04/2004 9:30	8260W	
439102	W	5430-0204-EW3-003	02/03/2004 9:30	03/04/2004 9:30	8330Prep	
439102	W	5430-0204-EW3-003	02/03/2004 9:30	03/04/2004 9:30	8330W	
439102	W	5430-0204-EW3-003	02/03/2004 9:30	03/04/2004 9:30	160.2WTSS	
439102	W	5430-0204-EW3-003	02/03/2004 9:30	03/04/2004 9:30	3010A	Mn Fe
439102	W	5430-0204-EW3-003	02/03/2004 9:30	03/04/2004 9:30	6010	Mn Fe
439103	W	5430-0204-EW4-004	02/03/2004 9:23	03/04/2004 9:23	3010A	Mn Fe
439103	W	5430-0204-EW4-004	02/03/2004 9:23	03/04/2004 9:23	6010	Mn Fe
439103	W	5430-0204-EW4-004	02/03/2004 9:23	03/04/2004 9:23	160.2WTSS	
439103	W	5430-0204-EW4-004	02/03/2004 9:23	03/04/2004 9:23	8260W	
439103	W	5430-0204-EW4-004	02/03/2004 9:23	03/04/2004 9:23	8330Prep	
439103	W	5430-0204-EW4-004	02/03/2004 9:23	03/04/2004 9:23	8330W	

QAP Figure 7.5
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Analytical Management Laboratories - Sample Status and Receipt Report

AML Project Number	4391	**Client AML ID**	ECC
AML Profile Number	218	**Client Project ID**	FNOP Mead NE

AML Sample	*Matrix*	Client Sample ID	Date Collected	Projected Due Date	Procedure	Comments
439104	W	5430-0204-EW5-005	02/03/2004 11:40	03/04/2004 11:40	8330Prep	
439104	W	5430-0204-EW5-005	02/03/2004 11:40	03/04/2004 11:40	8330W	
439104	W	5430-0204-EW5-005	02/03/2004 11:40	03/04/2004 11:40	160.2WTSS	
439104	W	5430-0204-EW5-005	02/03/2004 11:40	03/04/2004 11:40	3010A	Mn Fe
439104	W	5430-0204-EW5-005	02/03/2004 11:40	03/04/2004 11:40	6010	Mn Fe
439104	W	5430-0204-EW5-005	02/03/2004 11:40	03/04/2004 11:40	8260W	
439105	W	5430-0204-EW6-006	02/03/2004 11:08	03/04/2004 11:08	8260W	
439105	W	5430-0204-EW6-006	02/03/2004 11:08	03/04/2004 11:08	6010	Mn Fe
439105	W	5430-0204-EW6-006	02/03/2004 11:08	03/04/2004 11:08	3010A	Mn Fe
439105	W	5430-0204-EW6-006	02/03/2004 11:08	03/04/2004 11:08	160.2WTSS	
439105	W	5430-0204-EW6-006	02/03/2004 11:08	03/04/2004 11:08	8330Prep	
439105	W	5430-0204-EW6-006	02/03/2004 11:08	03/04/2004 11:08	8330W	
439106	W	5430-0204-EW7-007	02/03/2004 11:00	03/04/2004 11:00	3010A	Mn Fe
439106	W	5430-0204-EW7-007	02/03/2004 11:00	03/04/2004 11:00	8260W	
439106	W	5430-0204-EW7-007	02/03/2004 11:00	03/04/2004 11:00	8330Prep	
439106	W	5430-0204-EW7-007	02/03/2004 11:00	03/04/2004 11:00	8330W	
439106	W	5430-0204-EW7-007	02/03/2004 11:00	03/04/2004 11:00	6010	Mn Fe
439106	W	5430-0204-EW7-007	02/03/2004 11:00	03/04/2004 11:00	160.2WTSS	

QAP Figure 7.5
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Analytical Management Laboratories - Sample Status and Receipt Report

AML Project Number	4391	**Client AML ID**	ECC
AML Profile Number	218	**Client Project ID**	FNOP Mead NE

AML Sample	*Matrix*	Client Sample ID	Date Collected	Projected Due Date	Procedure	Comments
439107	W	5430-0204-EW8-008	02/03/2004 10:05	03/04/2004 10:05	8260W	MS MSD
439107	W	5430-0204-EW8-008	02/03/2004 10:05	03/04/2004 10:05	8330Prep	MS MSD
439107	W	5430-0204-EW8-008	02/03/2004 10:05	03/04/2004 10:05	8330W	MS MSD
439107	W	5430-0204-EW8-008	02/03/2004 10:05	03/04/2004 10:05	160.2WTSS	
439107	W	5430-0204-EW8-008	02/03/2004 10:05	03/04/2004 10:05	3010A	MS MSD , Mn Fe
439107	W	5430-0204-EW8-008	02/03/2004 10:05	03/04/2004 10:05	6010	MS MSD, Mn Fe
439108	W	5430-0204-2EW8-009	02/03/2004 10:05	03/04/2004 10:05	8260W	
439108	W	5430-0204-2EW8-009	02/03/2004 10:05	03/04/2004 10:05	8330Prep	
439108	W	5430-0204-2EW8-009	02/03/2004 10:05	03/04/2004 10:05	8330W	
439108	W	5430-0204-2EW8-009	02/03/2004 10:05	03/04/2004 10:05	160.2WTSS	
439108	W	5430-0204-2EW8-009	02/03/2004 10:05	03/04/2004 10:05	3010A	Mn Fe
439108	W	5430-0204-2EW8-009	02/03/2004 10:05	03/04/2004 10:05	6010	Mn Fe
439109	W	5430-0204-EW9-011	02/03/2004 10:52	03/04/2004 10:52	160.2WTSS	
439109	W	5430-0204-EW9-011	02/03/2004 10:52	03/04/2004 10:52	3010A	Mn Fe
439109	W	5430-0204-EW9-011	02/03/2004 10:52	03/04/2004 10:52	6010	Mn Fe
439109	W	5430-0204-EW9-011	02/03/2004 10:52	03/04/2004 10:52	8330Prep	
439109	W	5430-0204-EW9-011	02/03/2004 10:52	03/04/2004 10:52	8260W	
439109	W	5430-0204-EW9-011	02/03/2004 10:52	03/04/2004 10:52	8330W	

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Analytical Management Laboratories - Sample Status and Receipt Report

AML Project Number	4391	**Client AML ID**	ECC
AML Profile Number	218	**Client Project ID**	FNOP Mead NE

AML Sample	*Matrix*	Client Sample ID	Date Collected	Projected Due Date	Procedure	Comments
439110	W	5430-0204-EW10-012	02/03/2004 10:45	03/04/2004 10:45	6010	Mn Fe
439110	W	5430-0204-EW10-012	02/03/2004 10:45	03/04/2004 10:45	8260W	
439110	W	5430-0204-EW10-012	02/03/2004 10:45	03/04/2004 10:45	8330Prep	
439110	W	5430-0204-EW10-012	02/03/2004 10:45	03/04/2004 10:45	8330W	
439110	W	5430-0204-EW10-012	02/03/2004 10:45	03/04/2004 10:45	160.2WTSS	
439110	W	5430-0204-EW10-012	02/03/2004 10:45	03/04/2004 10:45	3010A	Mn Fe
439111	W	GCW1-INF-022004	02/03/2004 13:45	03/04/2004 13:45	8260W	
439112	W	2GCW1-INF-022004	02/03/2004 13:45	03/04/2004 13:45	8260W	
439113	W	GCW1-EFF-022004	02/03/2004 13:50	03/04/2004 13:50	8260W	
439114	W	GCW2-INF-022004	02/03/2004 11:55	03/04/2004 11:55	8330Prep	
439114	W	GCW2-INF-022004	02/03/2004 11:55	03/04/2004 11:55	8330W	
439115	W	2GCW2-INF-022004	02/03/2004 11:55	03/04/2004 11:55	8330Prep	
439115	W	2GCW2-INF-022004	02/03/2004 11:55	03/04/2004 11:55	8330W	
439116	W	GCW2-EFF-022004	02/03/2004 12:05	03/04/2004 12:05	8330Prep	
439116	W	GCW2-EFF-022004	02/03/2004 12:05	03/04/2004 12:05	8330W	
439117	W	Trip Blank	02/03/2004 0:00	03/04/2004 0:00	8260W	

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Figure 7.6

SW-846 Method 5035 Sample Preservation Report

Analytical Management Laboratories
15130 South Keeler, Suite B, Olathe, KS 66062 Tel: (913) 829-0101 Fax: (913) 829-1181

FORM-C VOC Soil Sample Collection and Preservation Record per AML Plan C (SW-846 Method 5035)

CLIENT PROJECT & NO: Massey Rd DO# 0059

AML WORK ORDER NO: 4398

Date (sample collection): (field use only) 02-04-04

Date (log-in): (lab use only) 02-05-04

Field Sample Number (field use only)	Vial Number (field use)	Preservative (ml) A = Acid & M = Methanol (field use)	Weight (VP, g) Of Vial (V) + preservative (P) (field use)	Weight (FW, g) of V + P + sample (S) (field use)	Weight (LW, g) of V + P + sample (S) (lab use only)	Wt. Diff. (FW-LW) (lab use only)	Sample (S) Weight (g) (FW-VP) (lab use)	Lab. Sample Number (lab use only)	Comments (lab use only)
FBSB-90-SS-1	A	A	32.86		37.47		4.61	4398-06	
	B	A	32.97		36.81		3.84	4398-06	
	C	M	32.63		38.41		5.78	4398-06	
FBSB-90-SB-1	A	A	33.05		37.99		4.94	4398-07	
	B	A	32.66		41.13		8.47	4398-07	
	C	M	32.79		39.78		6.99	4398-07	
FBSB-90-SS-2	A	A	33.25		39.87		6.62	4398-08	
	B	A	33.14		38.85		5.71	4398-08	
	C	M	32.70		40.48		7.78	4398-08	
FBSB-90-SB-2	A	A	32.92		41.16		8.24	4398-09	
	B	A	32.76		40.08		7.32	4398-09	
	C	M	32.70		39.36		6.66	4398-09	
FBSB-90-SS-3	A	A	33.02		40.61		7.59	4398-10	
	B	A	32.84		38.30		5.46	4398-10	
	C	M	32.81		38.98		6.17	4398-10	
FBSB-90-SS-4	A	A	32.97		38.21		5.24	4398-11	
	B	A	32.60		39.10		6.50	4398-11	
	C	M	33.01		38.79		5.78	4398-11	
FBSB-90-SB-4	A	A	32.92		38.34		5.42	4398-12	
	B	A	33.10		39.56		6.46	4398-12	
	C	M	32.80		38.93		6.13	4398-12	
FBSB-90-SS-5	A	A	32.96		37.19		4.23	4398-13	
	B	A	32.86		38.68		5.82	4398-13	
	C	M	32.98		39.32		6.34	4398-13	
FBSB-90-SB-5	A	A	33.11		39.20		6.09	4398-14	
	B	A	33.05		41.11		8.06	4398-14	
	C	M	32.77		37.95		5.18	4398-14	

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Analytical Management Laboratories

15130 South Keeler, Suite B, Olathe, KS 66062 Tel: (913) 829-0101 Fax: (913) 829-1181

FORM-A VOC Soil Sample Collection and Preservation Record per AML Plan A (SW-846 Method 5035)

CLIENT PROJECT & NO: Ft. L. Wood

AML WORK ORDER NO: 4458

Date (log-in): 02-20-04

Date of methanol extraction: (when applicable)

Field Sample Number	Lab. Sample Number	Vial Number	Weight of vial only (g)	Wt. of vial Plus sample (g)	Sample Wt. (grams)	Preservation F = Freezing & M = Methanol	Comments (lab use only)
OA1 XBKG-2	4458-01	A	27.07	31.26	4.19	F	
		B	27.30	32.33	5.03	F	
		C	27.05	32.14	5.09	F	
VOC XBKG-2	4458-01	D	26.93	31.51	4.58	F	
		E	27.03	31.75	4.72	F	
		F	27.28	31.65	4.37	F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	

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QAP Figure 7.7

Figure 7.7

Internal Chain of Custody Record

Analytical Management Laboratories - Internal Chain of Custody

AML Project Number 4466 **Client AML ID** CESAS

AML Profile Number 2123 **Client Project ID** Donaldson T43, DO# 0064

AML Sample	*Matrix*	Client Sample ID	Analysis Request	Removed Date/Time	Removed Analyst	Replaced Date/Time	Replaced Analyst	Removed Date/Time	Removed Analyst	Replaced Date/Time	Replaced Analyst
446601	W	DI-MW-1202-2-04	EPA 300.0 Water								
446602	W	DI-T43-GP-1D-2-04	EPA 300.0 Water								
446603	W	DI-T43-DUP-2-04	EPA 300.0 Water								
446604	W	DI-T43-PP-2-2-04	EPA 300.0 Water								
446605	W	DI-W-27-87-2-04	EPA 300.0 Water								
446606	W	DI-T43-PP-1-2-04	EPA 300.0 Water								
446607	W	DI-T43-GP-2-2-04	EPA 300.0 Water								
446608	W	DI-T43-PP-3-2-04	EPA 300.0 Water								
446609	W	DI-T43-PP-4-2-04	EPA 300.0 Water								
446610	W	DI-T43-PP-7-2-04	EPA 300.0 Water								
446611	W	DI-T43-PP-5-2-04	EPA 300.0 Water								
446612	W	DI-T43-BLK1-2-04	EPA 300.0 Water								

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QAP Fig 7.8
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Figure 7.8

AML LIMS Sample Preparation Batching Document

BATCHED SAMPLE WORKLIST
January 12, 2004 13:49

Queue MDIG Batch Rule 3015A Created 01/12/04 Analyst A Petrosino
Batch Number 1791 HBN 5765

	HSN	Procedure	Analyte List	Due Date	CC	Exp Date	Client	Turn	SamTyp	Orig HSN	Mgr	Collected	%Mos	Project ID
1	15034	3015A	3015A	01/14/04 13:49		07/10/04	In-house QC Account	W005	MB		amy			
2	15035	3015A	3015A	01/14/04 13:49		07/10/04	In-house QC Account	W005	LCS		amy			
3	15036	3015A	3015A	01/14/04 13:49		07/10/04	In-house QC Account	W005	LCSD		amy			
4	428601	3015A	3015A	01/13/04 00:24		07/04/04	US Army Corps of Engi	R014	SAMPLE			01/06/04		Forbes S-6
5	428602	3015A	3015A	01/13/04 00:24		07/04/04	US Army Corps of Engi	R014	SAMPLE			01/06/04		Forbes S-6
6	428603	3015A	3015A	01/13/04 00:24		07/04/04	US Army Corps of Engi	R014	SAMPLE			01/06/04		Forbes S-6
7	428604	3015A	3015A	01/13/04 00:24		07/04/04	US Army Corps of Engi	R014	SAMPLE			01/06/04		Forbes S-6
8	428605	3015A	3015A	01/13/04 00:24		07/04/04	US Army Corps of Engi	R014	SAMPLE			01/06/04		Forbes S-6
9	15037	3015A	3015A	01/09/04 10:00		07/04/04	In-house QC Account	R014	MS	428605	amy	01/06/04		
10	15038	3015A	3015A	01/09/04 10:00		07/04/04	In-house QC Account	R014	MSD	428605	amy	01/06/04		
11	15039	3015A	3015A	01/09/04 10:00		07/04/04	In-house QC Account	R014	PDS	428605	amy	01/06/04		
12	15040	3015A	3015A	01/09/04 10:00		07/04/04	In-house QC Account	R014	SD	428605	amy	01/06/04		
13	428606	3015A	3015A	01/13/04 00:24		07/04/04	US Army Corps of Engi	R014	SAMPLE			01/06/04		Forbes S-6
14	428607	3015A	3015A	01/13/04 00:24		07/04/04	US Army Corps of Engi	R014	SAMPLE			01/06/04		Forbes S-6
15	428609	3015A	3015A	01/14/04 03:59		07/05/04	US Army Corps of Engi	R014	SAMPLE			01/07/04		Forbes S-6
16	428610	3015A	3015A	01/14/04 03:59		07/05/04	US Army Corps of Engi	R014	SAMPLE			01/07/04		Forbes S-6
17	428611	3015A	3015A	01/14/04 03:59		07/05/04	US Army Corps of Engi	R014	SAMPLE			01/07/04		Forbes S-6

-------LEGEND-------

3015A - EPA 3015A Water Prep for 6020

3015A - 3015A List

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QAP Fig 7.9
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Figure 7.9

AML LIMS Sample Analysis Batching Document

BATCHED SAMPLE WORKLIST
January 13, 2004 08:36

Queue META Batch Rule 6020A Created 01/13/04 Analyst J Preheim
Batch Number 1740 HBN 5773

	HSN	Procedure	Analyte List	Due Date	CC	Exp Date	Client	Turn	SamTyp	Orig HSN	Mgr	Collected	%Mos	Project ID
1	15037	6020A	6020W ALL	01/11/04 10:00		07/04/04	In-house QC Account	R014	MS	428605	amy	01/06/04		
2	15038	6020A	6020W ALL	01/11/04 10:00		07/04/04	In-house QC Account	R014	MSD	428605	amy	01/06/04		
3	15039	6020A	6020W ALL	01/11/04 10:00		07/04/04	In-house QC Account	R014	PDS	428605	amy	01/06/04		
4	15040	6020A	6020W ALL	01/11/04 10:00		07/04/04	In-house QC Account	R014	SD	428605	amy	01/06/04		
5	15034	6020A	6020W ALL	01/16/04 13:49		07/10/04	In-house QC Account	W005	MB		amy			
6	15035	6020A	6020W ALL	01/16/04 13:49		07/10/04	In-house QC Account	W005	LCS		amy			
7	15036	6020A	6020W ALL	01/16/04 13:49		07/10/04	In-house QC Account	W005	LCSD		amy			
8	428601	6020A	6020W ALL	01/18/04 14:48		07/04/04	US Army Corps of Engi	R014	SAMPLE			01/06/04		Forbes S-6
9	428602	6020A	6020W ALL	01/18/04 14:48		07/04/04	US Army Corps of Engi	R014	SAMPLE			01/06/04		Forbes S-6
10	428603	6020A	6020W ALL	01/18/04 14:48		07/04/04	US Army Corps of Engi	R014	SAMPLE			01/06/04		Forbes S-6
11	428604	6020A	6020W ALL	01/18/04 14:48		07/04/04	US Army Corps of Engi	R014	SAMPLE			01/06/04		Forbes S-6
12	428605	6020A	6020W ALL	01/18/04 14:48		07/04/04	US Army Corps of Engi	R014	SAMPLE			01/06/04		Forbes S-6
13	428606	6020A	6020W ALL	01/18/04 14:48		07/04/04	US Army Corps of Engi	R014	SAMPLE			01/06/04		Forbes S-6
14	428607	6020A	6020W ALL	01/18/04 14:48		07/04/04	US Army Corps of Engi	R014	SAMPLE			01/06/04		Forbes S-6
15	428609	6020A	6020W ALL	01/19/04 18:23		07/05/04	US Army Corps of Engi	R014	SAMPLE			01/07/04		Forbes S-6
16	428610	6020A	6020W ALL	01/19/04 18:23		07/05/04	US Army Corps of Engi	R014	SAMPLE			01/07/04		Forbes S-6
17	428611	6020A	6020W ALL	01/19/04 18:23		07/05/04	US Army Corps of Engi	R014	SAMPLE			01/07/04		Forbes S-6

-------LEGEND-------

6020A - EPA 6020A Water

6020W ALL - 6020W ALL List

QAP Fig 7.5
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QAP Fig. 7.10
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Figure 7.10

AML Sample Disposal Record

Analytical Management Laboratories
Sample Disposal Record

ical Management Laboratories

Project	HSN	Client	Client Sample	Date Collected	Date Received	Expected Disposal Date	Matrix	Disposal Method / Disposed As: Trash Dumpster	Soil Hazwaste Drum	PCB Waste	Non-PCB Oil Product	CL Solvent Waste	Acid /Metals Drum	Mercury Waste	GC Vials Drum	Misc Lab Pack	Basic Waste Drum	Lab Sink / City Sewer	Return to Client	Consumed in Analysis
4481	448101	Tetra Tech	HER-2545	2/19/04	2/23/04	4/23/04	S	–	–	–	–	–	–	–	–	–	–	–	–	–
4481	448102	Tetra Tech	HER-2546	2/19/04	2/23/04	4/23/04	S	–	–	–	–	–	–	–	–	–	–	–	–	–
4481	448103	Tetra Tech	HER-2547	2/19/04	2/23/04	4/23/04	S	–	–	–	–	–	–	–	–	–	–	–	–	–

Date Disposed _____ Disposed By _____ Approved By _____

QAP Fig 7.10
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QAP Fig 12.1
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Figure 12.1

Example Test-Specific Checklist
Explosives Analysis

QAP Fig 12.1
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EXPLOSIVES ANALYSIS (HPLC) REVIEW CHECKLIST

Analytical Management Laboratories

Analyst: _____ Date: _____ Prep Batch No: _____
Reviewer 1 _____ Date: _____ Anal Batch No: _____
Reviwer2: _____ Date: _____ AML WO #: _____

ITEM NO. Main	Sub	DESCRIPTION OF ITEM CHECKED	CRITERIA	Yes	No	COMMENTS USACE Criteria: C*
I		**Instrument Calibration**				Includes initial calibration and its verification for both primary and confirmation columns.
	1	**Initial calibration-Primary column**				**List outlier compounds**
		Perfomed at min. 5 concentration levels?				
		%RSD for each compound < criteria?	20%			
	C*	Max %RSD for any compound < criteria?	40%			
		Mean of %RSD for all compounds <20%?	20%			
		Linear Regression (LR) performed?				
		Corr. Coeff. For LR >0.990?				
		Attached ICAL Summary and printouts?				
	2	**Initial calibration-Confirmation column**				**List outlier compounds**
		Perfomed at 5 concentration levels?				
		%RSD for each compound < criteria?	20%			
	C*	Max %RSD for any compound < criteria?	40%			
		Mean of %RSD for all compounds <20%?	20%			
		Linear Regression (LR) performed?				
		Corr. Coeff. For LR >0.990?				
		Attached ICAL Summary and printouts?				
	3	**Calibration Verification (CALV#1)**				**List outlier compounds**
		Primary: %D for each compound <15%	15%			
		Confirm: %D for each compound <15%	15%			
		Primary: Mean %D for all cpds. <15%?				
		Confirm: Mean %D for all cpds. <15%				
	C*	Max %D for any compound < criteria?	30%			
		Attached Summary and printouts?				
	4	**Calibration Verification (CALV#1)**				**List outlier compounds**
		Primary: %D for each compound <15%	15%			
		Confirm: %D for each compound <15%	15%			
		Primary: Mean %D for all cpds. <15%?				
		Confirm: Mean %D for all cpds. <15%				
	C*	Max %D for any compound < criteria?	30%			
		Attached Summary and printouts?				
	5	**Calibration Verification (CALV#1)**				**List outlier compounds**
		Primary: %D for each compound <15%	15%			
		Confirm: %D for each compound <15%	15%			
		Primary: Mean %D for all cpds. <15%?				
		Confirm: Mean %D for all cpds. <15%				
	C*	Max %D for any compound < criteria?	30%			
		Attached Summary and printouts?				

COMMENTS AND NOTES:

_____

_____

_____

_____

_____

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Page 3 of 3

EXPLOSIVES ANALYSIS (HPLC) REVIEW CHECKLIST

Analytical Management Laboratories

Analyst: _____ Date: _____ Prep Batch No: _____
Reviewer 1 _____ Date: _____ Anal Batch No: _____
Reviwer2: _____ Date: _____ AML WO #: _____

ITEM NO. Main	Sub	DESCRIPTION OF ITEM CHECKED	CRITERIA	Yes	No	COMMENTS
						USACE Criteria: C*
II		**Prep Batch Specific QC Elements**				**To be completed for each matrix**
	1	**Sample extraction log present?**				**Applies to each QC prep batch**
		Reviewed and initialed prep log?				
		Attached a copy of the prep. Log?				
	2	**Percent moisture/solids log present?**	**Not Applicable**			NOTE:Air-dried soil samples are used for analysis.
	3	**Method Blank**				**Applies to target compounds**
		Compounds if present, within QC limits?	0.5*MQL			
	C*	%R for water surrogate within QC limits?	60-140%			
	C*	%R for soil surrogate within QC limits?	50-150%			
		Form-I prepared and attached?				
		Instrument printouts attached?				
	4	**LCS and LCSD (if applicable)**				
		LCS recoveries within QC limits?	DoD QSM			
		LCSD recoveries within QC limits?	DoD QSM			DoD QSM Criteria for marginal exceedance (ME)
		RPDs for LCS/LCSD within QC limits?	50%			
	C*	%R for water surrogate within QC limits?	60-140%			
	C*	%R for soil surrogate within QC limits?	50-150%			
		Flagged (*) exceptions?				
		Form-I and III prepared and attached?				
		Instrument printouts attached?				
	5	**MS and MSD (if applicable)**				**Applies to spiked compounds**
		MS recoveries within QC limits?	DoD QSM			
		MSD recoveries within QC limits?	DoD QSM			DoD QSM Criteria for marginal exceedance (ME)
		RPDs for MS/MSD within QC limits?	50%			
		Flagged (*) exceptions?				
		Form-I and III prepared and attached?				
		Instrument printouts attached?				
III		**AML Project Specific QC Elements**				**To be completed for each AML project**
	1	**Project Info / Holding times (days)**				
		Sampling dates (COC/log-in) available?				
		Special QC requirements, if any?				
		Holding times (W&S) for extraction met?	7&14			
		Holding times for analysis met?	40			
	2	**Sample Results Calculations**				**Applies to each sample in the project**
		Surrogate recoveries within limits?	50-150%			
		If not, reanalysis performed?				
		Sample reanalyzed if above ICAL limit?				
		Sample dilution factors correct?				
		MDLs, RLs, MQLs adjusted for dilution?				
		Primary: Positives within RT window?				
		Confirm: Positives within RT window?				
		RPD for each positives <40%?	40%			
		Checked for anomalies if RPD is >40%?				
		Data qualified if RPD is >40%?				
		Results below MQL qualified as J?				
		Form-I prepared and attached?				
		Instrument printouts attached?				

2006 AML Standard Operating Procedures

STANDARD OPERATING PROCEDURES

No. O-MEE-RC

ANALYSIS OF METHANE, ETHANE AND ETHENE (MEE) IN AQUEOUS SAMPLES BY HEADSPACE SAMPLING AND GAS CHROMATOGRAPHY/FID (GC/FID) (EPA RSK-175 and EPA SW846-8015B mod.)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure SOP) is used to determine petroleum gases such as methane, ethane, and ethene or ethylene (MEE) in aqueous samples. This SOP incorporate the SOP developed by EPA Robert S. Kerr (RSK) Groundwater Research Laboratory located in Ada, OK.

1.2 The method detection limits (MDLs) and quantitation limits (MQLs) for MEE have been determined in our laboratory. These are shown in Table 1.

1.3 The instrument employed at Analytical Management Laboratories (AML) is a Hewlett Packard 5890 GC with a flame ionization (FID) detector.

1.4 The terms, SOP, procedure, and method are used interchangeably in this document. This method is based upon the theories of headspace sampling.

1.5 This draft AML SOP is restricted to experienced analytes in the use of these theories and skilled in the interpretation of chromatograms and their use as a quantitative tool.

1.6 The SOP for RSK-175 method is attached in Appendix 1.

1.7 Technical description of the method is attached in Appendix 2.

---------- End of Narrative ----------

Table 1
Aqueous Sample MDL, MQL, and QC Limits for LCS and MS

Methane/Ethane/Ethene (MEE) Analysis by GC/FID														
Analytical Management Laboratories														
Matrix: Water	Preparation Method			5021/RSK 175										
	Analytical Method			8015/RSK 175										
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**		**LCS Limits**		**MS Limits**		**RPD**
Compound	Amount	ug/L	ug/L	**ug/L**	RD=B/A	flag	**ug/L**	RQ=C/A	flag	Lower	Upper	Lower	Upper	Limit
	ug/L	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%
Methane	5	3.57	0.48	**1.51**	2.4		**4.4**	2.9	*	70	130	70	130	30
Ethane	5	8.20	0.61	**1.91**	4.3		**9.2**	4.8		70	130	70	130	30
Ethene	5	7.52	0.47	**1.49**	5.1	*	**13.6**	9.1		70	130	70	130	30
Mean Ratio (RD or RQ):					3.9			5.6						
MDL= ST Dev * t factor (3.14 for 7 reps)														
Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 5.														
Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.														
LCS and MS %R limits are laboratory limits.														

Appendix 1

RSKSOP-175, Revision 0

Appendix 2

Technical Description of the RSKSOP-175 method

STANDARD OPERATING PROCEDURES

No. O-OCPesticides-RC

ORGANOCHLORINE PESTICIDES BY GC/ECD (EPA Methods 508.1, 608 and SW846-8081B)

October 14, 2004

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP), which is based on EPA methods (508.1, 608, SW-846 8081A and 8081B-Draft Revision 2A) is used to determine the concentrations of various organochlorine pesticides in solid and liquid matrices, using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD). The compounds listed in Table 1 may be determined by either a single- or dual-column analysis system.

1.2 This SOP, which is based on EPA 508.1 and 608 and a revision of EPA SW-846 Method 8081 no longer includes Polychlorinated Biphenyls (PCBs) as Aroclors in the list of target analytes. The analysis of PCBs should be performed using AML SOP based on SW-846 Method Method 8082, which includes specific cleanup and quantitation procedures designed for PCBs analysis. Therefore, when performing analysis for 508.1 and 608 analytes, use AML SOP based on Method 8082 for PCBs analyses, and this SOP for the quantitative analysis of organochlorine pesticides and screening for PCBs.

1.3 If there is no information of the likely presence of PCBs, either employ a PCB-specific screening procedure such as an immunoassay (e.g., SW-846 Method 4020), or split the sample extract *prior to* any cleanup steps, and process part of the extract for organochlorine pesticide analysis and the other portion for PCB analysis using AML SOP based on Method 8082.

1.4 Surrogates are identical for the two methods and the sample preparation procedures are similar. Therefore, sample extracts and the method blank extract can be split prior to performing cleanup procedures that are specific for each analysis. Separate laboratory control samples and matrix spike samples should be analyzed for each method.

1.5 Several multi-component mixtures (i.e., Chlordane and Toxaphene) are listed as target analytes. When samples contain more than one multi-component analyte, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of multi-component analytes that have been subjected to environmental degradation or degradation by treatment technologies. These result in "weathered" multi-component mixtures that may have significant differences in peak patterns than those of standards.

1.6 This SOP describes anaylsis that employs a hardware configuration of two analytical columns joined to a single injection port. One injection is used for dual-column analysis.

1.7 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs (GC) and skilled in the interpretation of gas chromatogram.

1.8 This SOP may be used to analyze for additional compounds, which are shown in Table 2. The target list of compounds for specific projects may include one or more compounds from Table 2 in addition to the default list of compounds shown in Table 1.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample (approximately 1-L for liquids, 2-g to 30-g for solids) is extracted using appropriate matrix-specific sample extraction techniques.

2.2 Liquid samples are extracted at neutral pH with methylene chloride using procedures described in this SOP or AML SOPs based on SW-846 Method 3510C (separatory funnel), SW-846 Method 3520C (continuous liquid-liquid extractor), solid-phase extraction (Method 3535A) or other appropriate technique. The SW-846 methods including the overview (Method 3500B) should be used for additional guidance on cleanup procedures.

2.3 Solid samples are extracted with methylene chloride-acetone (50:50) using AML SOPs based on Method 3540C (Soxhlet), Method 3541 (automated Soxhlet), Method 3545 (pressurized fluid extraction), Method 3550B (ultrasonic extraction), microwave extraction (Method 3546) supercritical fluid extraction (Method 3562) or other appropriate technique. The SW-846 methods including the overview (Method 3500B) should be used for additional guidance on cleanup procedures.

2.4 This SOP is intended primarily for low-level definitive laboratory analysis of aqueous and soil samples for organochlorine pesticides using procedures shown in Sections 2.2 and 2.3. With appropriate modifications, microextraction procedures (SW-846 Methods 3511/3570) may also be used to support rapid turnaround analysis to support environmental remediation efforts. The microextraction procedures that are described in this SOP are similar to those described in SW-846 method 8011 and EPA Method 504.1.

2.5 A variety of cleanup steps may be applied to the extract, depending on the nature of the matrix interferences and the target analytes. Suggested cleanup procedures include AML SOPs based on SW-846 methods that employ alumina (Method 3610B and 3611B), Florisil (Method 3620B), silica gel (Method 3630C), gel permeation chromatography (Method 3640A), and sulfur (Method 3660B). The SW-846 methods including the overview (Method 3600C) should be used when a suitable AML SOP is unavailable during method development and/or implementation.

2.6 After cleanup, the extract is analyzed by injecting a 1-µL sample into a gas chromatograph with a narrow- or wide-bore fused silica capillary column and electron capture detector (GC/ECD).

3.0 INTERFERENCES

3.1 Interferences can be caused by a number of sources in the extraction and analyses stages. Quality control measures, such as the analysis of calibration blanks, method blanks, and solvent blanks can be used to monitor for and determine the interference.

3.2 Contamination may occur when a high-concentration and a low concentration sample are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. A high concentrated sample should be followed by the analysis of solvent to check for possible cross contamination.

3.3 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination should be expected with sediment samples. AML SOPs based on SW-846 Method 3660B is suggested for removal of sulfur. Since the recovery of Endrin aldehyde (using the TBA procedure) is drastically reduced, this compound must be determined prior to sulfur cleanup. The SW-846 methods should be used when a suitable AML SOP is unavailable during method development and/or implementation.

3.4 Halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. Certain co-eluting organophosphorus pesticides are eliminated by AML SOPs based on Method 3640A (gel permeation cleanup - pesticide option). Co-eluting chlorophenols may be eliminated by using Method 3630 C(silica gel), Method 3620B (florisil), or Method 3610B (alumina). Polychlorinated biphenyls (PCBs) also may interfere with the analysis of the organochlorine pesticides. The problem may be most severe for the analysis of multicomponent analytes such as Chlordane, Toxaphene, and Strobane. If PCBs are known or expected to occur in samples, the analyst should consult AML SOPs based on SW-846 Methods 3620B and 3630C for techniques that may be used to separate the pesticides from the PCBs. The SW-846 methods should be used when a suitable AML SOP is unavailable during method development and/or implementation.

3.5 The following compounds may coelute using the dual-column analysis scheme. In general, the DB-5 column resolves fewer compounds that the DB-1701.

DB-5	Permethrin/Heptachlor epoxide
	Endosulfan I/α-Chlordane
	Perthane/Endrin
	Endosulfan II/Chloropropylate/Chlorobenzilate
	4,4'-DDT/Endosulfan sulfate
	Methoxychlor/Dicofol
DB-1701	Chlorothalonil/β-BHC
	δ-BHC/DCPA/Permethrin
	α-Chlordane/*trans*-Nonachlor

Nitrofen, Dichlone, Carbophenothion, Dichloran exhibit extensive peak tailing on both columns. Simazine and Atrazine give poor responses on the ECD detector.

Triazine compounds should be analyzed using AML SOPs based on Method 8141 (NPD option) or the original SW-846 method.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph – Two Hewlett Packard (Agilent) 5890 Series II gas chromatographs (GCs) are used. The systems are complete with a temperature-programmable GC for splitless injection, all accessories, including syringes, analytical columns and gases, and Agilent 7673 or 7673A autosamplers. The column flow rate remains constant throughout temperature program operation.

4.2 Gas chromatographic columns – The instrument is operated in the dual column mode. The following GC capillary columns are employed:

4.2.1 A 30 meter x 0.32 mm ID capillary column coated with DB-5 (J&W Scientific) with a film thickness of 0.25 um.

4.2.2 A 30 meter x 0.32 mm ID capillary column coated with DB-1701 (J&W Scientific) with a film thickness of 0.25 um.

4.2.3 Other equivalent columns may be used in place of both columns.

4.3 Data system - Hewlett Packard's Environquant is used to allow continuous acquisition and storage on machine-readable media of all data obtained throughout the duration of the GC program.

4.4 Microsyringes - 10, 25, 100, 250, 500 and 1000 uLs.

4.5 Balance - Analytical, 0.0001 grams and top loading, 0.1 grams.

4.6 Disposable pipettes - Pasteur

4.7 Test tubes - 5 ml to 8 ml, with Teflon lined screw caps.

4.8 Separatory funnels - 2000 mL

4.9 Kuderna-Danish (K-D) apparatus a sdescribed below:

4.8.1 KD Tubes

4.8.2 KD Evaporation flask (500 mls), attached to concentrator tubes

4.8.3 Three ball macro snyder column

4.8.4 Clamps

4.10 Boiling Chips - solvent extracted

4.11 Heated water bath

4.12 Tekmar Sonic Disrupter

4.13 Glass wool

4.14 Glass beads, solvent-rinsed, bake in 400□C oven for approximately one hour

4.15 Stainless steel table spoons

4.16 IKA Automated Soxhlet Extractors – Six 4-position units equipped with heating devices, refrigerated circulating coolers, personal computer for instrument control, and micron filters to hold samples.

4.17 Soxtec Automated Soxhlet Extractors – Three 6-position units (HT-6) and two 2-position units, equipped with temperature-controlled oil bath (Soxtec). Accessories for the Soxtec equipment will include the following: Tecator bath oil (catalog number 1000-1886), contamination-free cellulose extraction thimbles (26 mm ID x 60 mm, catalog number 1522-0034), glass extraction cups (80 mL, catalog number 1000-

18200, thimble adapters (catalog number 1000-14660 and Viton seals (catalog number 1000-2516).

4.18 Circulating refrigerated coolers

4.19 Mechanical shakers

4.20 40-mL VOA vials (or equivalent) for microextraction of aqueous samples (SW-846 Method 3511) for rapid turnaround analysis (in support of remediation efforts)

4.21 Polytetrafluoroethylene (PTFE) tubes (25-mL capacity) for microscale solvent extraction (SW-846 Method 3570) of soil and waste samples for rapid turnaround analysis (in support of remediation efforts)

4.22 Mechanical centrifuge

4.23 Injector vials

4.24 Sonic Disrupter - Tekmar Model TM500 Sonic Disruptor with 500-watt power and pulsing capabilities. Use with ¾" horns and for guidance on use, see AML SOP on sonicator tuning and maintenance.

4.25 Tekmar Model V1A 3/4" Horn (probe).

5.0 REAGENTS

5.1 It is assumed that all inorganic/organic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where available. Any other reagents must demonstrate high purity without lessening the accuracy of the determination.

5.2 Organic free reagent water - All references to water in this method will be assumed to be organic free reagent water, except when referring to field samples.

5.3 Sulfuric acid solution

5.3.1 Slowly add 50 mL of sulfuric acid to 50 mL of water (1:1 v/v)

5.4 Solvents

5.4.1 Acetone – GC grade

5.4.2 Hexane – GC grade

5.4.3 Methylene chloride - GC grade

5.5 Stock Standard solutions - Available from Ultra Scientific (Catalog number PPM-808C). The list of analytes and concentrations are shown in in Appendix 1. Stock surrogate standards are also available from Ultra (catalog # ISM-320).

5.6 GC/ECD tuning standard solution. This solution, which is available from Supelco (Cat # 4-8397) contains DDT at 100ng/ml and Endrin at 50ng/ml. Prepare standard by diluting using 0.50ml of the commercial standard to 50ml of acetone.

5.7 Calibration Standards - Prepare a 100-ml stock standard solutions at 1000 ng/ml from Ultra Scientific catalog log number PPM-808C (20 compounds - conc. 1000ug/ml), & Ultra Scientific catalog number ISM-320 (TCMX & DCB 200 ug/ml) . Take 100ul of PPM-808C (Pesticides Compounds) & 0.500 of ISM-320 (TCMX & DCB) and dilute up to 100ml with acetone. The calibration curve consist of five points: 10ng/ml , 30ng/ml, 50ng/ml, 70ng/ml, & 100ng/ml. Preparation is as follows: 10ng/ml- (0.5ml of stock diluted to 50ml with acetone), 30ng/ml-(1.5ml of stock diluted to 50ml with acetone), 50ng/ml-(2.5ml of the stock diluted to 50ml with acetone), 70ng/ml-(3.5 ml of the stock diluted ot 50ml with acetone), 100ng/ml-(5.00ml of the stock diluted to 50ml with acetone).

5.8 Sample surrogate spiking solution - The sample surrogate spiking solution is purchased as a shooter from Ultra-Scientific at 200 ng/ml cat. # ISM-321X. Spike 1.0 mL of the surrogate solution into each sample including QC samples. This solution contains decachlorobiphenyl and tetrachloro-m-xylene.

5.9 Matrix Spike (MS) spiking solution is prepared from a second source stock standard solution (Accustandard), which contains each target compound at a concentration of 1.0-mg/mL. Dilute 250-uL of thhis solution to 250-mL with acetone. The resulting matrix spike solution is at a concentration of 1000 ng/ml. Spike each MS and MSD sample with 0.250-ml of the 1000-ng/mL standard.

5.10 Laboratory Control Sample (LCS) spiking solution is the same as the matrix spike stnadard. Spike each sample with 0.25-mL of the 1000-ng/mL spiking solution.

5.11 Sodium Hydroxide Solution (10N) - Dissolve 40 grams of sodium hydroxide solution into 100 mLs of reagent water.

5.12 Solvents used in the extraction and cleanup procedures (AML SOPS based on appropriate 3500 and 3600 series methods) include n-hexane, diethyl ether, methylene chloride, acetone, ethyl acetate, and isooctane (2,2,4-trimethylpentane). The solvent must be exchanged to n-hexane or isooctane prior to analysis. Soil Sample should be extracted in a 50%Acetone/50 % Methylene Chloride solution**.**

5.13 Separate calibration standards are required for each multi-component target analyte (e.g., Toxaphene and Chlordane). The analysts should evaluate the Toxaphene standard carefully. Some Toxaphene components, particularly the more heavily chlorinated components, are subject to dechlorination reactions. As a result, standards from different vendors may exhibit marked differences which could lead to possible false negative results or to large differences in quantitative results.

5.14 Internal standard. The compound, 1-bromo-2-nitrobenzene is used as an internal standard for the dual-column analysis. Prepare an intermediate solution of 1-bromo-2-nitrobenzene (50-ug/L) by diluting 1.0-mL of the commercial stock solution (Ultra, cat # PPS-351, 5,000-ug/mL) to 100-mL with acetone or iso-octane. Dilute 10-mL of this intermediate solution (50,000-ng/mL) to 100-mL with acetone to prepare the working standard at a concentration of 5,000-ng/mL. Spike 10 µL of this solution into each 1 mL of sample extract and calibration solutions. The final concentration of the internal standard in sample extracts and standards should be 50 ng/mL.

5.15 Sodium sulfate, granular, anhydrous (Na_2SO_4) for drying samples and sample extracts, ACS reagent grade.

6.0 SAMPLE PRESERVATION HANDLING & STORAGE

6.1 One litre amber glass bottles with Teflon lined screw caps may be used for collecting aqueous samples in accordance with EPA SW-846 method 8081A. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 2 bottles for each water sample to be collected. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.

6.2 One 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples in accordance with EPA SW-846

method 8081A. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each solid sample. Quality control samples such as MS and MSD should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. These samples may be shared with samples collected for other analyses such as semivolatiles (8270C), PCBs (8082), Diesel (DRO, 8015-mod.) and other pesticides (8140, etc.).

6.3 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Analytical data generated outside of the specified holding times must be considered to be minimum values only. Such data may be used to demonstrate that a waste is hazardous where it shows the concentration of a constituent to be above the regulatory threshold but cannot be used to demonstrate that a waste is not hazardous. The holding time for the extraction of aqueous samples, which includes TCLP leachates is 7 days from the date of sample collection or TCLP leachate preparation. The holding times for the extraction of soil, sediment and waste samples, which includes TCLP leachate preparation is 14 days from the date of sample collection. The holding time for the instrumental analysis of sample extracts is 40 days from the date of extraction.

7.0 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control contain a number of regulated compounds at high concentrations. The toxicity of these compounds present in such complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Aqueous and soil samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters after the solvent(s) have been allowed to evaporate in a hood.

9.0 SAMPLE EXTRACTION

9.1 Preparation of Sediment and Soil Samples

9.1.1 Sample Extraction by Sonication – SW-846 Method 3550

9.1.1.1 Weight out 30-g of the soil sample to three significant numbers (±0.1-g) into 500-mL beakers. Record the exact weight in the organic Extraction logbook (See Appendix 11 for documentation). Add enough sodium sulfate (60-g) to dry the soil and mix well with a spoon. When the soil is dry (freely flowing powder), add 1.0-mL of surrogate solution to all the samples (including QC) and 0.25-ml of the appropriate spiking solutions to the LCS/LCSD & MS/MSD.

9.1.1.2 Add 100-mL of the solvent mixture (methylene chloride:acetone 50:50 v/v) to the beaker and mix the contents with a spoon for 1 minute. Place the bottom surface of the ¾" horn about ½" below the surface of the solvent, but above the sediment layer. The horn should be properly tuned in accordance with the manufacturer instructions (see AML SOP on sonicator tuning). Extract ultrasonically for 3 minutes, with output control knob set at 10 (full power) and with mode switch on Pulse (pulsing energy rather than continuous energy) and percent-duty cycle knob set at 50% (energy on 50% of time and off 50% of time). Verify that the soil is free flowing and not in clumps and that the slurry has a smooth rolling action with each pulse of energy. Decant the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus.

9.1.1.3 Extract the sample twice more with 100-mLs of methylene chloride/acetone solvent mixture. The solution should be sonicated for 3 minutes each time. Decant the solution in to the same K-D as in the previous step.

9.1.1.4 Add two boiling chips to the K-D flask and attach a three-ball Snyder column. Prewet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1-ml, add 5-mL of hexane and continue with solvent removal. When the extract reaches 1 to 2-mL remove the K-D flask and allow to cool for at least 10 minutes.

9.1.1.5 Quantiatively transfer the extract to a 10-mL culture tube equipped with a PTFE-lined cap with graduation at 5-mL. Adjust the final volume to 5-mL with hexane. Proceed to analyze by GC/ECD if no cleanup procedures are required. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for

instrumental analysis so that the internal standard concentration is 50-ng/µL.

9.1.2 Sample Extraction by Automated Soxhlet Procedure A – SW-846 Method 3541

9.1.2.1 Automated Soxhlet Extraction Procedure-A involves IKA equipment, which are capable of handling 30-g sample aliquots. The set-up consists of six 4-position extractors to handle a total of 24 samples, which will include up to 5 QC samples (MB, LCS, LCSD, MS, and MSD). The six extractors, equipped with independent heating and cooling systems are controlled by a single personal computer. Follow manufacturer instructions for setting up and operating the IKA Soxhlet equipment.

9.1.2.2 Weight out 30-g of the soil sample to three significant numbers (±0.1-g) into 500-mL beakers. Record the exact weight in the organic Extraction logbook (See Appendix 11 for documentation). Add enough sodium sulfate (60-g) to dry the soil and mix well with a spoon. When the soil is dry (freely flowing powder), the sample is ready for Soxhlet extraction.

9.1.2.3 Clean and completely assemble the IKA equipment. Raise the condenser from the top vessel. Assemble the top vessel with the stainless steel filter base and a new disposable Teflon micron-sized filter paper. Transfer the soil/drier mixture quantitatively to the top vessel. Add 1.0-mL of surrogate solution to all the samples (including QC) and 0.25-mL of the appropriate spiking solutions to the LCS/LCSD & MS/MSD samples. Add 100-mL of the solvent mixture (methylene chloride:acetone 50:50) to the bottom vessel and tightly screw the top vessel on to the bottom vessel containing solvent. Replace the condenser unit on the top vessel. Turn the unit on and allow the unit to cycle (45-minutes). Completely drain the solvent from the sample chamber using the instrument cycle control mechanism. Repeat the extraction process twice using the same solvent contained in the bottom vessel. Decant the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus.

9.1.2.4 Add two boiling chips to the K-D flask and attach a three-ball Snyder column. Prewet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1-ml, add 5-mL of hexane and continue with solvent removal. When the extract reaches 1 to 2-mL remove the K-D flask and allow to cool for at least 10 minutes.

9.1.2.5 Quantiatively transfer the extract to a 10-mL culture tube equipped with a PTFE-lined cap with graduation at 5-mL. Adjust the final volume to 5-mL with hexane. Proceed to analyze by GC/ECD if no cleanup procedures are required. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 50-ng/µL.

9.1.3 Sample Extraction by Automated Soxhlet Procedure B – SW-846 Method 3541

9.1.3.1 Automated Soxhlet Extraction Procedure-B involves Tecator Soxtec equipment, which are capable of handling only 10-g sample aliquots. The set-up consists of three 6-position extractors and two 2-position extractors to handle a total of 22 samples, which will include up to 5 QC samples (MB, LCS, LCSD, MS, and MSD). The five extractors are operated independent using three hot oil baths equipped with independent controllers. The condensers are cooled using a circulating refrigerant bath. Follow manufacturer instructions for setting up and operating the Soxtec equipment.

9.1.3.2 Weight out 10-g of the soil sample to three significant numbers (±0.1-g) into 500-mL beakers. Record the exact weight in the organic Extraction logbook (See Appendix 11 for documentation). Add enough sodium sulfate (10-g) to dry the soil and mix well with a spoon. When the soil is dry (freely flowing powder), transfer the entire contents of the beaker quantitatively to the rigid paper thimbles suspended from the stainless steel holders. Add 1.0-mL of surrogate solution to all the samples (including QC) and 0.25-mL of the appropriate spiking solutions to the LCS/LCSD & MS/MSD samples.

9.1.3.3 Clean and completely assemble the Soxtec extractors. Check the heating oil level in the automated Soxhlet unit and add oil if needed. See service manual for details. Set the temperature on the service unit at 80°C. Press the “MAINS” button and verify that the switch lamp is now in the “ON” position. Set the flow rate for the circulator at approximately 2-L/min to minimize solvent loss from the condensing unit.

9.1.3.4 Immediately transfer the thimbles containing the weighed samples into the condensers. Raise the knob to the "BOILING" position. The magnet will now fasten to the thimble. Lower the knob to the "RINSING" position. The thimble will now hang just below the condenser valve.

9.1.3.5 Insert the extraction cups containing boiling chips, and load each with 50-mL of extraction solvent. Using the cup holder, lower the locking handle, ensuring that the safety catch engages. The cups are now clamped into position. (The seals must be pre-rinsed or pre-extracted with extraction solvent prior to initial use.)

9.1.3.6 Move the extraction knobs to the "BOILING" position. The thimbles are now immersed in solvent. Set the timer for 60 minutes. The condenser valves must be in the "OPEN" position. Extract for the preset time.

9.1.3.7 After rinse time has elapsed, close the condenser valves by turning each a quarter-turn, clockwise. When all but 2- to 5-mL of solvent have been collected, open the system and remove the cups.

9.1.3.8 Transfer the contents of the cups to 15-mL graduated, conical-bottom glass tubes attached to the K-D flasks. Rinse the cups using methylene chloride and add the rinsates to the glass tubes.

9.1.3.9 Add two boiling chips to the K-D flask and attach a three-ball Snyder column. Pre-wet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1-ml, add 5-mL of hexane and continue with solvent removal. When the extract reaches 1 to 2-mL remove the K-D flask and allow to cool for at least 10 minutes.

9.1.3.10 Quantiatively transfer the extract to a 10-mL culture tube equipped with a PTFE-lined cap with graduation at 5-mL. Adjust the final volume to 5-mL with hexane. Proceed to analyze by GC/ECD if no cleanup procedures are required. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 50-ng/µL.

9.1.4 Microscale Solvent Extraction (SW-846 Method 3570) – This procedure should be used only in support of remediation efforts and for projects requiring relatively high detection limits. The Project Manager for specific projects may approve this extraction procedure.

9.1.4.1 Weight out 2.5-g (±0.5-g) of the soil sample to three significant numbers (±0.01-g) into 25-mL PTFE vials which has a PTFE screw cap. Record the exact weight in the organic Extraction logbook (See Appendix 11 for documentation). Add enough sodium sulfate (2.5-g) to dry the soil and 5 to 10 pre-cleaned glass beads for mixing the contents. Wipe the lips and threads of the tube with paper towels.

9.1.4.2 Add 1.0-mL of surrogate solution to all the samples (including QC) and 0.25-mL of the appropriate spiking solutions to the LCS/LCSD & MS/MSD samples. Add 12-mL of the extraction solvent to the tube and cap tightly. Shake the tubes vigorously until the slurry is free flowing. Use a mechanical shaker or vortex mixer, if necessary, breaking up any chunks with a metal spatula.

Add more anhydrous sodium sulfate and manually mix as necessary to produce a free-flowing, finely divided slurry.

9.1.4.3 Extract the samples using a mechanical shaker or a rotating end-over-end unit for four hours. Decant or pipet the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus. Extract the soil sample in the PTFE tube twice with approximately 5-mL of the extraction solvent and shaking vigorously by hand for two minutes. Rinse the sodium sulfate with 2 to 3-mL of the extraction solvent.

9.1.4.4 Add two boiling chips to the K-D flask and attach a three-ball Snyder column. Pre-wet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1-ml, add 5-mL of hexane and continue with solvent removal. When the extract reaches 1 to 2-mL remove the K-D flask and allow to cool for at least 10 minutes.

9.1.4.5 Quantiatively transfer the extract to a 10-mL culture tube equipped with a PTFE-lined cap with graduation at 5-mL. Adjust the final volume to 5-mL with hexane. Proceed to analyze by GC/ECD if no cleanup procedures are required. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 50-ng/µL.

9.2 Preparation of Water Samples

9.2.1 Manual Liquid-Liquid Extraction (SW-846 Method 3510C)

9.2.1.1 Using a black marker, mark the meniscus of the water in the sample container. Pour the entire contents of the container into a 2-liter separatory funnel. Rinse the sample container with 3 aliquots of 20-mls methylene chloride and place the rinse in the separatory funnel. Carefully add tap water to the sample container up to the marked meniscus. Pour the distilled water into a 1-liter graduated cylinder. Record this measured volume in the organochlorine pesticides Extraction logbook (See Appendix 11 for documentation).

9.2.1.2 An alternate procedure, which may be employed to measure out water samples is described below. Using a 1-Liter graduated cylinder, measure out 1.00 liter ± 0.005 Liters (or lower volumes as low as 100-mL for high concentration samples) of the sample, and transfer it to a 2000-mL separatory funnel. Record the exact volume in the organic Extraction logbooks. This alternate

procedure may be used to measure out lower sample volumes for the re-extraction of heavily contaminated samples with surrogate recoveries outside the QC limits, which are diluted out during analysis. This procedure may also be used to measure out smaller aliquots (e.g. 500-mL) of the sample for MS/MSD analysis, when additional sample containers were not collected for these analyses. NOTE: The measuring cylinder must be thoroughly cleaned using soap and water, rinsed with copius amounts of tap water, deionized water, and acetone prior to using it to measure out another environmental sample.

9.2.1.3 Using pH indicating paper (0-14 for general purpose and a narrow range paper 4 to 10 range for pH adjustment) record the pH of the sample (sample should be at a neutral pH). This is accomplished by dipping the tip of a Pasteur pipette into the water and placing the droplets of water on the pH paper. Never place the pH paper into the water sample. Adjust, if necessary, the pH to 7 using either 50/50 sulfuric acid solution or 10N sodium hydroxide solution. Add 1.0-mL of surrogate solution to all of the samples (including QC) and 0.25-mL of the appropriate spiking solution to the LCS/LCSD & MS/MSD.

9.2.1.4 Add 60-mLs of methylene chloride to the separatory funnel and shake for 2 minutes. Take care to vent the separatory funnel to relieve excess pressure. Allow the organic layer to separate from the water layer for a minimum of 10 minutes. Drain the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus. Repeat the extraction 2 more times (total 3 extractions) using 60-mLs of methylene chloride. Combine the extracts into a single KD vessel.

9.2.1.5 Add two boiling chips to the K-D flask and attach a three ball Snyder column. Prewet the Snyder column with 1 mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1-ml, add 5-mL of hexane and continue with solvent removal. When the extract reaches 1 to 2-mL remove the K-D flask and allow to cool for at least 10 minutes.

9.2.1.6 Quantiatively transfer the extract to a 10-mL culture tube equipped with a PTFE-lined cap with graduation at 5-mL. Adjust the final volume to 5-mL with hexane. Proceed to analyze by GC/ECD if no cleanup procedures are required. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 50-ng/µL.

9.2.2 Microextraction (SW-846 Method 3511) - This procedure should be used only in support of remediation efforts and for projects requiring relatively high detection limits. The Project Manager for specific projects may approve this extraction procedure. Samples may be collected in 40-mL VOA vials for this method if relatively volatile analytes are also included analysis using this determinative procedure. If they are collected in 1-L amber bottles, an aliquot may be transferred to a 40-mL VOA vial (including two additional aliquots for the sample selected for MS/MSD samples). Use reagent water contained in 40-mL vials for MB, LCS, and LCSD analyses. Use alternate pre-cleaned glass vials equipped with PTFE-lined caps, if the centrifuge does not accommodate 40-mL VOA vials.

9.2.2.1 Remove the cap from the VOA vial and use a disposable pipette to remove approximately 5-mL of water. Work with a single field or QC sample at a time and label the vial and the cap to minimize cross-contamination. Weigh the capped vial to the nearest 0.1-g and record it in the logbook. **NOTE:** Re-weigh the empty vial when the sample extraction procedure is completed (after completing Section 9.2.2.5) or weigh the vial before using it for microextraction. The difference between the two weights (g) should be used as sample volume (mL) assuming a density of 1.0 for aqueous samples.

9.2.2.2 Use a surrogate spiking solution (in acetone) that is 10-times more concentrated than those used for other extraction procedures (see Section 5.8) to maximize recoveries. Spike with 100-µL of the surrogate solution (10-times lower volume than for other extraction methods).

9.2.2.3 For spiked sample analysis, use LCS or MS spiking solutions (in acetone) that is 10-times more concentrated than those used for other extraction procedures (see Sections 5.9 and 5.10) to maximize recoveries. Spike with 25-µL of the LCS/MS spike solution (10-times lower volume than for other extraction methods).

9.2.2.4 Add exactly 2.0-mL (**NOTE:** This volume (2-mL) is the final extract volume for this procedure although half this volume is used for instrumental analysis) of methylene chloride using a gas-tight syringe or a class A volumetric pipette and approximately 12-g of anhydrous sodium chloride to the VOA vials. Replace the cap. Shake the vials vigorously for five minutes or until the sodium chloride dissolves completely. The agitation may be performed using a mechanical shaker, if necessary. Briefly allow the phases to settle, then centrifuge at 500 times the gravity for 15-minutes.

9.2.2.5 Tilt the VOA vial to collect the lower methylene chloride to one section of the vial bottom. Pack a small pasteur pipette with glasswool and 100-mg of anhydrous sodium sulfate. Using a 1-mL

gas-tight syringe, transfer approximately 1.5-mL of the lower methylene chloride layer through the pasteur pipette to a K-D tube. Add 1-mL of hexane through the pasteur pipette and concentrate using nitrogen blow-down to a small volume. When the extract reaches approximately 1-ml, add 1-mL of hexane and continue with solvent exchange.

9.2.2.6 Adjust the fnal volume to 1.0-mL of the dried extract in an injection vial. Proceed to analyze by GC/ECD if no cleanup procedures are required. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 50-ng/µL.

9.3 Sample Preparation for waste samples

9.3.1 If the waste is soluble in extraction solution, add approximately 1.0-g to a 15-mL culture tube. Record the exact weight in the Organic Extraction Log. Add 1.0-mL of the surrogate solution for samples and 1.0mL of surrogate plus 0.25-mL of appropriate spiking solution for LCS/LCSD & MS/MSD samples.

9.3.2 Dilute the extract to 10.0 mLs with extraction solution and proceed with GC/ECD analysis. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 50-ng/µL.

9.3.3 If the waste is not soluble in the extraction solution, the sample must be extracted like a sediment/soil sample using only 1.0-g instead of 30-g. Proceed to cleanup steps if the sample appears to be dirty.

9.4 Extract Cleanup Procedures

Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements. General guidance for sample extract cleanup is provided in this section, in AML SOPs based on SW-846 3600 Series methods or the SW-846 methods themselves. The SW-846 methods should be used when a suitable AML SOP is unavailable during method development and/or implementation.

9.4.1 The SOP based on SW-846 Method 3620 (florisil) is used as the deafult cleanup procedure by AML to separate organochlorine pesticides from aliphatic compounds, aromatics, and nitrogen-containing compounds. Most if not all the samples and associated QC samples are subjected to this cleanup procedure prior to instrumental analysis.

9.4.2 If a sample is of biological origin, or contains high molecular weight materials, the use of SOPs based on Method 3640 (GPC cleanup - pesticide option) should be used. Frequently, one of the adsorption chromatographic cleanups (alumina, silica gel or Florisil) may also be required following the GPC cleanup.

9.4.3 AML SOPs based on SW-846 Method 3610 (alumina) may be used to remove phthalate esters.

9.4.4 AML SOPs based on SW-846 Method 3630 (silica gel) may be used to separate single component organochlorine pesticides from some interferants.

9.4.5 Elemental sulfur, which may be present in certain sediments and industrial wastes, interferes with the electron capture gas chromatography of certain pesticides. Sulfur should be removed by the technique described in AML SOPs based on SW-846 Method 3660.

9.4.6 Use the SW-846 method when a suitable AML SOP is unavailable during method development and/or implementation.

9.5 INSRUMENTAL ANALYSIS PROCEDURE

9.5.1 Dual-column analysis. The dual-column/dual-detector approach involves the use of two 30 m x 0.53 mm ID fused-silica open-tubular columns of different polarities, thus, different selectivities towards the target analytes. The columns are connected to an injection tee and separate electron capture detectors.

9.5.1.1 Retention times for the organochlorine analytes on the GC columns employed are shown in the instrument printouts, which are included in Appendix 2.

9.5.1.2 Intial temp – 80 degree

9.5.1.3 Hold time – 2.0 minute

9.5.1.4 3.0 degree/minute 250 degrees, hold for 2.0 minute.

9.5.1.5 Multi-component mixtures of Toxaphene and Chlordane are analyzed separately using the same conditions as above.

9.5.2 Tuning Solultion - DDT and Endrin are easily degraded in the injection port. Breakdown occurs when the injection port liner is contaminated high boiling residue from sample injection or when the injector contains metal fittings. Check for degradation problems by injecting a standard containing only 4,4'-DDT and Endrin. Presence of 4,4'-DDE, 4,4'-DDD, Endrin ketone or Endrin indicates breakdown. If degradation of either DDT or Endrin exceeds 15%, take corrective action before proceeding with calibration.

9.5.2.1 Calculate percent breakdown as follows:

$$\%\ \text{breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD + DDE)}}{\text{sum of all peak areas (DDT + DDE + DDD)}} \text{x}\,100$$

$$\% \text{ breakdown of Endrin} = \frac{\text{sum of degradation peak areas (aldehyde + ketone)}}{\text{sum of all peak areas (Endrin + aldehyde + ketone)}} \text{x} 100$$

9.5.2.2 The breakdown of DDT and Endrin should be measured before samples are analyzed. Injector maintenance and recalibration should be completed if the breakdown is greater than 15% for either compound. The calculation of DDT and Endrin breakdown is illustrated in Appendix 2.

9.5.3 Once the criteia shown above has passed, the initial calibration can be analyzed. Directly inject the initial calibration solution containing the single component pesticides in the following order: 10 STD, 30 STD, 50 STD, 70 STD, & 100 STD. Analyze a mid-level standard of the multi-component pesticides such as technical chlordane and toxaphene (oe Strobane, if required) immediately after the analysis of a set of ICAL solutions.

9.5.4 The calibration curve for single component pesticides may be run from high to low instead of low too high as decribed. Summarize the initial calibration data in Form-5 equivalents (See Appendix 3). After the completion of the 50 STD standard ensure that all compounds have been appropriately identified. The analyst may choose to run a six point calibration, but it is not required by this method, but a five point is. One point may be dropped from the calibration if necessary, but not the low point, which is used to establish MQLs.

9.5.5 Tabulate the area response of the characteristic ions against concentration for each compound and each standard. Calculate response factors (RF) for each compound relative to the internal standards. For multi-component pesticides, use either the total area or 3 to 5 (4 to 6 for some programs) major peaks. The RF calculation is:

$$RF = (A_s C_{is}) / (A_{is} C_s)$$

where:

A_s = Area of the characteristic ion for the compound being measured
A_{is} = Area of the characteristic ion for the specific internal standard
C_{is} = Concentration of the specific internal standard
C_s = Concentration of the compound being measured

9.5.6 The average RF (RF_m) should be calculated and recorded for each compound using the five or six RF values calculated from the initial (5or 6-point) calibration curve. Using the RFs from the initial calibration, calculate and record the percent relative standard deviation (%RSD) and the mean response factor (RF_m) for each compound. The percent RSD is calculated as follows:

$$\% \text{ RSD} = ((SD) * 100\%/(RF_m)$$

where:

RF_m = mean of the 5 or 6 initial RFs for a compound
SD = standard deviation of the 5 or 6 initial RFs for a compound.

9.5.7 Linearity – If the % RSD of any single-component pesticide compound is 20% or less, then the relative response factor is assumed to be constant over the calibration range and the average relative response factor may be used for quantitation. It is desirable to review for trends as a function of the concentration for all compounds and exercise professional judgment to determine use of regression methods even for compounds that meet the <20% RSD criteria as described in the following section. Additionally, it is recommended that the fit of individual calibration points to the calibration line or curve be visually examined to determine if different calibration technique should be used. Since regression methods normally weigh more toward high-level standards, weighed regression methods are recommended if the concentration of concern is near low-level standards.

9.5.8 If the % RSD of any detected compound is greater then 20%, use least square regression methods to calculate sample concentrations. For regression methods, the instrument response is treated as the dependent variable (y) and the standard amount is the independent variable (x). When "Cs" is used as "x", use "As*Cis/Ais" as "y". When "Cs/Cis" is used as "x", use "As/Ais" as "y". Do not include origin (0,0) as an extra calibration point.

9.5.9 Although weighted least squares method is preferred for calculating concentrations more accurately at lower levels, commercially available software (check with the instrument manufacturer) should be used to perform regression procedures. A linear least squares equation ($y = a*x + b$) is preferred over non-linear calibration equations ($y = a*x^2 + b*x + c$). The coefficient of determination (COD or r^2) or correlation coefficient (r) should be used to measure the goodness of fit. For regression models to be used for quantitative purposes, the COD or r^2 must be greater than or equal to 0.99 and the correlation coefficient (r) should be greater than 0.995. A value of 1.00 for COD and/or "r" indicates a perfect fit.

9.5.10 Corrective action may be required if the criteria for %RSD, r, r2, or COD are not met.

9.5.11 An initial calibration (ICAL) should be considered a single event process. Consult the QA office or check applicable EPA methods (e.g. SW-846 Method 8000C and 8081A) for additional guidance on appropriate corrective action.

9.5.12 Re-fitting of ICAL – The selected model for ICAL may be subjected to an additional re-fitting check to establish representativeness of the model for each compound in each ICAL standard. This is an optional procedure, which is recommended but not required since automated data processing procedures may not be able to accommodate it. Calculate the %D between

the calculated amount (Cc) for each compound in each ICAL standard from the ICAL model and its expected value (Ce). Ideally, the %D ((Cc-Ce) * 100 /Ce) for each compound in ICAL standard should meet the QC criteria for %RSD. It is possible for one or more ICAL standards to have %D exceeding the %RSD criteria, while the %RSD for ICAL model is within QC limits. The re-fitting procedure may be used to identify ICAL standards that need re-analysis or dropped from the calculation of ICAL model when re-analysis was not performed.

9.5.13 Check and optimize instrument operating conditions including automated peak integration procedures for appropriateness in addressing the chromatographic needs of the specific compound with problems.

9.5.14 If the problem appears to be associated with just one of the ICAL standards, that one standard may be reanalyzed once immediately (must be within 8-hours) after the ICAL. Discard all the results from the original analysis of the standard in question and replace it with the results from re-analysis.

9.5.15 When additional calibration standards (six or seven instead of five) are analyzed, narrowing the range of concentrations to be used for determining the goodness of fit is an acceptable procedure. Five or more standards should remain for the calculation of RSD or linear regression for each compound after one or more points are removed from the ICAL. More points (6 for second order and 7 for third order) may be needed for non-linear regression. It is generally prohibited to remove data points from within a calibration range, while still retaining the extreme ends of the calibration range. Do not discard a middle point unless it appears to have an obvious problem and six or more standards were analyzed for ICAL and re-analysis of the affected standard was not performed within the required time limits. Changes to the upper end of the calibration range will affect the need to dilute samples above the range. A change to the lower range is not recommended since it will affect the method quantitation limit (MQL) used to report data. The default MQLs, which are based on the lowest ICAL concentration are stored in the LIMS and project-specific changes are not practical.

9.5.16 The refitting procedures may lead to failed %Ds for upper level calibration standards for average response factor methods. The analyst may take alternate corrective actions such as the analysis of sample extracts under dilution for the affected compounds.

9.5.17 The refitting procedures may lead to failed %Ds for lower level calibration standards for regression methods. The analyst may take appropriate corrective action such as flagging the sample result as an estimated amount ("J" flag) at the lower level encountered in the sample.

9.5.18 Grand mean RSD – Previous versions of the EPA method(s) had provisions for the calculation of the grand mean %RSD for all target compounds in the ICAL solution, when the % RSD of any detected compound is greater than 20%. The grand mean %RSD should be <20% for ICAL to be acceptable. This procedure is discontinued. Criteria for Initial calibration are compound-specific.

9.5.19 The term "calibration verification" (CALV) is used to describe initial verification (ICV) of the initial calibration (ICAL) using second-source standards and subsequent continuing verifications (CCVs) using same-source standards. The response factors calculated during CALV are not used for sample quantitation since CALV standards are not used as "continuing calibration" standards permitted in some of the EPA methods.

9.5.20 Initial Calibration Verification (ICV) - Analysis of a second source ICV standard is required before any samples can be analyzed for USACE projects. Since second source standards are not employed for the analysis of LCS/LCSD and MS/MSD samples, ICV analysis is applicable for all projects. Calculate the percent difference (%D) or %Drift for the observed response factor in ICV from the mean RF as described for CCV. The %D or %drift should not exceed ±15% for each target compound. Document calibration verification in instrument printouts as shown in Appendix 2. Check the ICAL and ICV standards if it is not achievable for all the target compounds without any exception. Use professional judgment for non-USACE projects and for compounds that are not contaminants of concern (COC) for USACE projects. The ICV may be used as the beginning CCV for sample analysis.

9.5.21 If the above criteria are met, the analysis for analytical samples can begin.

9.5.22 Continuing Calibration Verification (CCV) – The initial calibration curve for each compound of interest must be checked and verified once every 12-hours with a calibration verification standard. Analyze the mid level STD and calculate the percent difference (%D) of the RRF from the average RRF for each compound. The %D for each compound should be <15%. If the %D for any compound is greater than 15%, the GC should be restored to proper operating condition before re-analyzing the CCV for all the compounds. A new ICAL should be genearted if the CCV does not meet QC criteria.

9.5.23 Grand mean %D – Previous versions of the EPA method(s) had provisions for the calculation of the grand mean %D for all target compounds in the CCV stanadard, when the %D of any detected compound is greater than 15%. The grand mean %D should be <15% for CCV to be acceptable. This procedure is discontinued. Criteria for CCV is compound-specific. Summarizes CCV in Form-7 equivalents (See Appendix 4).

9.5.24 Calibration Factors are used when external standards are used for calibration. This procedure is not used at AML and they are described only when they are necessary. Situations include internal standard coeluting with one of the traget compounds in one or more columns. When external standard calibration is employed, calculate the calibration factor for each analyte at each concentration, the mean calibration factor, and the relative standard deviation (RSD) of the calibration factors, using the formulae shown below. If internal standard calibration is employed, refer to Method 8000 for the calculation of response factors. Calculate the calibration factor for each analyte at each concentration as:

$$CF = \frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

Calculate the mean calibration factor for each analyte as:

$$\text{mean CF} = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

where n is the number of standards analyzed. Calculate the standard deviation (SD) and the RSD of the calibration factors for each analyte as:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF})^2}{n-1}} \qquad RSD = \frac{SD}{\overline{CF}} \times 100$$

If the RSD for each analyte is 20%, then the response of the instrument is considered linear and the mean calibration factor can be used to quantitate sample results. If the RSD is greater than 20%, then linearity through the origin cannot be assumed. The analyst must use a calibration curve or a non-linear calibration model (e.g., a polynomial equation) for quantitation. See SW-846 Method 8000C or AML SOPs (if available) based on this SW-846 Method for information on non-linear calibrations.

9.5.25 ANALYSIS OF SAMPLE EXTRACTS

9.5.25.1 Once the samples have been extracted and all system performance checks have been established as stated within this SOP, sample extracts can be analyzed. A 1-mL aliquot of the sample extract is spiked with 10-µLs of internal standard solution (1-bromo-2-nitrobenzene at 5000 ng/µL). The final concentration of internal standard is 50 ug/mL for each 1 mL extract.

9.5.25.2 If the response for any compound exceeds the initial calibration curve range of the GC/ECD system, extract dilutions must take place. Additional internal standard must be added to the diluted extract to maintain the required 50 ng/uL of each internal standard in the extract used for instrumental analysis. The diluted extract must be reanalyzed.

9.5.26 DATA INTERPRETATION

9.5.26.1 The qualitative identification of compounds determined by this method is based on retention time, and % difference between positive

results on the two columns. The qualitative identification of multi-component pesticides such as chlordane, toxaphene or strobane is made from their pattern and from the presence of characteristic peaks. Technical BHC and Technical DDT are not identified by their pattern. They are identified by their individual components (α-BHC, δ-BHC, DDD, DDE, etc.). Compounds should be identified as present when the criteria below are met.

9.5.26.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component in both GC columns. The calculated amount should be >MDL (or >LLR or >RL when these are used to define the lowest level for reporting positives) on both columns.

9.5.26.3 The relative percent difference (RPD) between the results obtained from the two columns should be <40%. If RPD is >40%, the result(s) should be reported as an estimated quantity with a flag (e.g. "J"). If one column result is significantly higher (e.g., >40%), check the chromatogram to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baselines for both columns and baseline parameters established for peak integrations. A rising baseline may cause a lower result. Manual integration may be necessary in some cases to obtain accurate peak areas. If no peak integration anomalies are noted, check chromatographic conditions and the chromatograms for LCS and LCSD samples for good separation of target analytes and reproducibility of results from the two chromatographic columns. If no anomalies are noted, matrix interference and/or the presence of non-target compounds may be inferred. Report result(s) in accordance with guidance provided in Section 9.5.26.4

9.5.26.4 Reporting of the results should follow method guidance and guidance from regulatory agencies (federal, state, local) and guidance from clients (contract requirements, QAPP requirements, etc.). Client requirements, when available should address regulatory requirements. AML guidance, which is based on SW-846 Method 8000C will serve as the deafult guidance as described below:

9.5.26.4.1 Default AML Procedure: Report the lower of the two results from the primary and confirmation columns. Report results with RPD >40% as estimated ("J" flag).

9.5.26.4.2 Client guidance and analyst discretion are very important. Some clients may require one of the columns to be designated as primary and the second column as confirmatory. In such cases, results may need to be reported from the primary column unless interference is suspected for a specific compound in the primary column, which are not present in the confirmation column.

9.5.26.4.3 The SW-846 Method 8000 guidance has not been consistent over time. Although the most recent Method 8000 revision (Rev. C) recommends the reporting of the lower result, earlier revisions (e.g. Rev. B) required the reporting of the higher result since it was considered to be more protective of the environment. Some EPA regional offices may have issued memoranda, which may require the reporting of the higher result.

9.5.26.4.4 Some projects may require the reporting of results from both columns.

9.5.26.5 Confirmation of positives may also be made from GC/MS analysis of the pesticide extracts when applicable and from the identification of the pesticide compound from BNA (SVOCs) fraction, if analyzed. Document sample result in worksheets that are Form-10 equivalents (See Appendix 6)

9.5.27 QUANTITATIVE ANALYSIS

9.5.27.1 The internal standard technique is used to determine the concentration of an identified target compound. For water samples the calculation is as follows:

$$\text{Concentration } (\mu g/L) = \frac{(A_s) * (IS) * (V_e)}{(A_{is})\,(RF_m)\,(V_s)}$$

where:

A_s = Area for compound being measured.
IS = Concentration of the internal standard in the extract injected (µg/mL)
Ais = Area for the internal standard
RF_m = Mean Response factor for compound being measured
V_s = Volume of water (L) extracted
V_e = Volume of the final extract (mL), taking into account any dilutions made

9.5.27.2 For soil samples the calculation is as follows:

$$\text{Concentration } (\mu g/kg) = \frac{(A_s) * (IS) * (V_e) * (100)}{(A_{is}) * (RF_m) * (W_s) * (\%S)}$$

where:

A_s = Area for compound being measured.
IS = Concentration of the internal standard in the extract injected (µg/mL)
Ais = Area for the internal standard
RF_m = Mean Response factor for compound being measured
W_s = Wet weight of soil sample (kg) extracted
V_e = Volume of the final extract (mL), taking into account any dilutions made

10.0 QUALITY CONTROL

10.1 Using this SOP will involve implementing the referenced EPA method at the laboratory. Method implementation involves the following: chromatographic separation of the target analytes on two GC columns, determination of retention time windows for each compound, determination of method detection limits for each sample matrix and/or preparation method, spiked sample analysis, initial demonstration of acceptable recoveries, etc. Method implementation procedures are described on the AML SOP on MDL.

10.2 Analytical data, which includes calibration data, sample preparation logs, sample data and quality control data should be gathered prior to review. Review checklist (See Appendix 5) should be used to document verification of calculations and adherence to method procedures. Exceptions should be documented. Samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on these checklists.

10.3 Summarize sample results using Form-1 Equivalents (See Appendix 12). Summarize QC results and QC recoveries in Form-1 and Form-3 equivalents respectively for MS/MSD (Appendix 7) and LCS/LCSD (Appendix 8).

10.4 Surrogate recovery should be within QC limits (DoD QSM, Revision 2, June 2002) for samples, spikes and blanks. Document surrogate recoveries in Form-2 equivalents (See Appendix 9) Flag recoveries that are outside the QC limits with an asterisk (*). If re-extraction and/or reanalysis has been performed, and the surrogate recoveries for reanalysis are within QC limits, report data based on acceptable surrogate recoveries. If the surrogate recoveries are outside the QC limits for the reanalysis, report data for both analyses with surrogate recovery flags.

10.5 Method blanks (MB) should be free of target compounds. The default QC limit for method blank is one half of the applicable MQL. See Tables 3 and 4 for a list of MDLs, LLRs, and MQLs. Details of the MDL determinations are included in Appendix 10. When project specific reporting limits are available, which may be greater than MQL, the project specific QC limit for MB is one half of the reporting limit. Samples associated with the method blank containing target compounds at levels greater than the QC limit will require re-extraction and reanalysis unless other criteria accepable to the client are used. Since method blanks may be contaminated when they are associated with samples containing high levels of target compounds, professional judgment must be exercised to determine samples requiring re-extraction and reanalysis.

10.6 LCS recoveries should be within QC limits (statistical control limits and DoD QSM limits) for the target compounds. Flag recoveries that are outside the quality control limits with “ME” and compounds outside the ME limits with “ME*”. See Tables 3 and 4 for QC limits. Contact supervisor for appropriate corrective action if more than the allowed number of compounds are outside the control limits or the marginal exccedance (ME) limits. Although data qualification may be sufficient for commercial clients, re-extraction, and/or reanalysis of associated samples will be

necessary for most DoD projects. Other constraints may include project schedule, turnaround time for re-analysis and the availability of additional sample volume.

10.7 MS recoveries should be within QC limits (DoD QSM) for the target compounds. Flag recoveries that are outside the control limits with "ME" and compounds outside the ME limits with "ME*". See Tables 3 and 4 for QC limits. Contact supervisor for appropriate corrective action if more than the allowed number of the compounds are outside the control limits or the marginal exccedance (ME) limits. Data qualification, re-extraction, and/or reanalysis of associated samples may be necessary depending on project requirements, turnaround time and other constraints.

10.8 Report sample results on Form-1 equivalents (See Appendix 12). Report target compounds that are present at levels greater than the reporting limit (RL) using "J" qualifiers if the levels are less than MQL. Compounds that were also detected in the associated MB should also be qualified with a "B" code. If the concentration of any of the target compounds exceed the upper limit of calibration (ICAL), flag the affected compounds data with an estimated or "E" flag. The extracts may need to be diluted and reanalyzed until the concentrations are within the calibration limits for normal turnaround analyses. For rapid turnaround analyses sample results may be reported with "E" qualifiers if sufficient time is not available for reanalysis.

11.0 **DATA VERIFICATION AND VALIDATION -** This section will provide guidance and procedures to assess if data produced using this SOP is valid and legally defensible. Document review and comments on the checklist for the raw data package or other detailedchecklists (See Appendix 5). Verify that sample preparation logs are complete and the instrument run logs are available for review (See Appendix 11). Anomalies and exceptions should be documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on the checklists. Army (USACE), Air Force (AFCEE) or Department of Defense (DoD) projects may have special requirements (See Appendix 13) that will require extra attention. A simple, universal procedure for evaluating and choosing appropriate corrective action doe not exist to deal with situations when one or more QC parameters are outside the acceptance window. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints.

11.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) projects may have additional requirements that are related to USACE laboratory validation (CX), USACE District requirements, government contract requirements, specific USACE project requirements or USACE Architect/Engineer (A/E) requirements. USACE-CX requirements (Summary of Method Quality Objectives for Method 8081A) and DoD requirements are included in Appendix 13.

11.1.1 The USACE requirements shown in Appendix 8 and elsewhere in this SOP are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the

legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may or may not be attainable for routine sample analysis. Since USACE MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

11.1.2 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality objectives and/or other QC requirements. Exceptions and data qualifications necessary should be documented on the checklists and eventually on case narratives.

11.1.3 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist, perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete. For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete. For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director will review the checklists (initial/date as necessary), prepare case narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

11.1.4 The blank criteria is one half of the applicable MQL (default criteria) or one half of the project specific reporting limit (which may be higher than MQL), whichever is applicable.

11.2HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

11.2.1 WATER - The holding times for semi-volatiles is 7 days from sampling.

11.2.2 SOIL – A 14 day holding is used for soil and waste samples.

11.2.3 TCLP – TCLP leachates must be extracted within 7 days after the TCLP extraction.

11.2.4 CHECK – Establish holding times by comparing the chain of custody sampling date with the date of the injection after purging. TCLP holding

times must be established by comparing first the COC to the leaching date and then the leaching date to the sample extraction date, followed by the extract injection date.

11.2.5 ACTION – If holding times have been exceeded, contact the client. All positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or innvalid (I).

11.3COLUMN PERFORMANCE – Column performance criteria for GC\ECD should meet criteria established in this SOP. Column performance is measured from the DDT breakdown to DDD and DDE and the endrin breakdown to its aldehyde and ketone (See Appendix 2 for details). Such breakdown should not exceed 15%. The breakdown should be checked every 12 hours.

11.3.1 Check to see if the column performance report is present and that it meet criteria above.

11.3.2 ACTION – If the breakdown is outside criteria, perform appropriate corrective action as described elsewher in this SOP and in the referenced SW-846 method, recalibrate and rerun the breakdown standard for an acceptable report. The calibration can not be run until the breakdown is in control. If full documentation on DDT/Endrin brekdown standard is not present, the package is considered incomplete.

11.4INITIAL CALIBRATION (**ICAL**)– Instrument calibration (See Appendix 3 for details) criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the GC/ECD is capable of producing data within a range that is linear. The ICAL includes initial verrification (ICV) of the ICAL with a second source standard for USACE and AFCEE projects (See Appendix 13 for details).

11.4.1 CHECK – Check to see if any other compounds are outside the criteria of 20% or less for %RSD. If a compound is outside the 20% criteria, check to see if regression methods are used. The goodness of fit should be measured with the coefficient of determination (COD) or related quantities (r^2 and r) greater than 0.99.

11.4.2 ACTION – Check and correct the standards, including the range of concantrations for appropriateness and repeat the ICAL analysis.

11.4.3 ACTION – Use a lesser number of peaks (minimum 3) if the criteria are not met for five peaks (desirable) selected for each multi-component analyte.

11.4.4 ACTION - Report the problem to the Project Manager and report detects and non-detects with aapropriate qualifiers for rapid TAT projects. Mention the ICAL anomaly in the case narrative.

11.5 INITIAL CALIBRATION VERIFICATION – Calibration verification includes initial calibration verification (ICV) and continuing calibration verifications (CCVs). A second source ICV is used to check the integrity of the ICAL standards used for calibrating the instrument. The 12-hour analytical shift begins with the injection of the ICV (instead of a CCV) for sample analyses that are performed immediately after an ICAL.

11.5.1 Target Compounds - Check to see if %D for any of the target compounds is outside the USACE QC criteria (<±15%). Check also for systematic errors that may result from bias between the two standards.

11.5.2 ACTION – When the problem is confined to a few specific target compounds, check peak separation, peak integration and integration parameters for anomalies. If no anomalies are found, check the ICAL and ICV standards for integrity. Replace one or more of the working standards (ICV first and ICAL later) for trouble shooting. Correct the problem(s) and repeat ICV analysis. Use professional judgment in selecting an appropriate corrective action for all projects.

11.5.3 ACTION - When a CCV immediately following the ICV is acceptable, peak integration and chromatograhic anomalies are not likely to be present. The analysis of standards from a third source may be necessary, if the inspection and replacement of ICV and ICAL standards fail to resolve the anomaly between ICAL and the second source ICV.

11.6 CONTINUING CALIBRATION VERIFICATION – The continuing calibration verification (See Appendix 4 for details) is used to check that the instrument is still operating under the linear range of the initial calibration and has remained stable every 12 hours.

11.6.1 IS area counts (use mean IS area from ICAL for the %D of IS area) must not vary by more than a factor of 2 (-50% or + 100%) from the associated ICAL. Check to see if the %D for any compounds are outside the criteria of 15% or less.

11.6.2 %D for Compounds present in the sample – The %D for the CCVs before and after the sample analysis should be within QC limits when internal standards are not used. The %D for the CCV before sample analysis should be within QC limits when internal stanadrds are used.

11.6.3 %D for Compounds not present in the sample – The %D for the CCVs before and after the sample analysis may be >(+15%). Corrective action is not required unless the %D is < (-15%).

11.6.4 ACTION – Corrective action must be performed for positive results with %D outliers. In exceptional cases (e.g rapid TAT analysis for screening level data), positive results for compounds not meeting the criteria may be qualified as estimated (J) by the user (client).

11.6.5 ACTION - Report non-detects without qualifiers and include a description of the anomaly in the case narrative.

11.6.6 ACTION – Perform more frequent CCVs (e.g every 10 samples) than required (every 12-hours) to avoid extensive re-analysis.

11.7METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Sample data qualification to alert blank contamination is performed by the laboratory. Sample data qualification based on all the blank results (laboratory MB, equipment blank, and other field blanks) is performed by the user (client). The processing of blanks helps eliminate reporting of false positives in project reports.

11.7.1 Method blanks should be free of all target anaytes and other interfering non-target analytes. To be acceptable, blank results should not exceed the QC limit, which is one half of the applicable MQL for any compound (default) or one half of the reporting limit (project specific).

11.7.2 Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples. If levels greater than the QC limit are present, inspect and correct the problem. Reanalyze all associated samples. Contact client if other criteria are applicable. The only exception may be quick turn around projects.

11.7.3 ACTION – All positive results are reported with a “B” code, if contaminants are found in the method blank.

11.7.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, then no action is taken.

11.7.5 ACTION – Any compound detected in the sample which were also detected in associated method blank at levels greater than the QC limit should be re-extracted unless other client criteria are applicable.

11.7.6 ACTION – If contaminants resulting in GC/ECD peak saturation are present, all affected compounds are qualified as invalid (I).

11.8SURROGATE RECOVERIES – Surrogates or system monitoring compounds are different structurally, but similar chemically to classes of target compounds. These surrogate compounds are added just prior to extraction of the samples and the percent recoveries of the surrogates are calculated and evaluated.

11.8.1 Verify that the surrogate recoveries (See Appendix 9 for details) for the surrogate compounds, DCB and TCMX are within the QC limits (laboratory control limits and DoD QSM control limits):

11.8.1.1 For aqueous samples, the DCB recoveries should be within the range, 30% to 135%. The TCMX recoveries should be within the range, 25% to 140%. Flag outliers with an asterisk (*).

11.8.1.2 For soil samples and other solid matrices, the DCB recoveries should be within the range, 55% to 130%. The TCMX

recoveries should be within the range, 70% to 125%. Flag outliers with an asterisk (*).

11.8.2 ACTION – When surrogate recoveries are outside control limits the sample must be re-analyzed. Reanalysis with or without cleanup may be performed under dilution, if necessary if original analysis should indicate high levels of target and non-target analytes.

11.8.3 ACTION - Reextraction and cleanup will be necessary if reanalysis does not resolve the problem. If surrogate recoveries are still outside the limits, report both sets of data and describe the problem in the case narrative.

11.9 LABORATORY CONTROL STANDARD (LCS) – The analysis of LCS samples is used to evaluate method competence in the absence of matrix effects. The LCS is evaluated by comparing the percent recovery for all of the target analytes to the laboratory QC limits, which are identical to the 3-sigma DoD QSM QC limits. See Appendix 10 for guidance on documenting LCS and LCSD concentrations and %R in QC summary report forms. The DoD QSM specifies the number of compounds that can exceed the 3-sigma QC limits based on statistical considerations. The LCS recovery for such compounds should not exceed the marginal exceedance 4-sigma (ME) limits. Evaluation procedures may include comparison of LCS recoveries with those determined by project-specific DQOs (See project QAPP, if available), laboratory statistical control limits, or USEPA guidance. Initially, the effect of QC failure on the samples should be evaluated. Regardless of this assessment, steps shall be taken to find the source of the problem and correct it.

11.9.1 CHECK - LCS and LCSD recoveries (See Appendix 8 for details) should be within QC limits (See Tables 3 and 4). Compare the %R results for each compound in LCS with laboratory QC limits to see if all the results are within the acceptable range. Flag outliers with “ME” if it is within the 3-sigma limits. Flag it with “ME*” if it is outside the 4-sigma ME limits.

11.9.2 CHECK - Compare observed %R results for each compound with the laboratory limits for LCS/LCSD recoveries (e.g. USACE criteria). Perform manual calculation for %R, if necessary for QC outliers. Document the number of compounds that exceeded the QC limit and compare it against the maximum recommended by DoD QSM. Document this review in checklists (Appendix 7) and in case narratives.

11.9.3 ACTION - Typically, the LCS would be reanalyzed for the failed analytes (ME*) only. If the second analysis fails, then the LCS, method blank, and all associated samples of the batch would be reanalyzed for the failed analytes only. If sufficient sample is not available for reanalysis, or if the corrective action is ineffective, discuss the situation with the client project manager (or chemist). Include a discussion of the situation in the case narrative. The case narrative should discuss the corrective action taken and any other information.

11.9.4 ACTION - For non-USACE projects, no action may need to be taken on LCS and/or LCSD recovery data alone either to reanalyze samples and/or to qualify associated samples. Providing data of known quality is

sufficient for most projects. If significant deviations (e.g. number of target compound exceeding QC limits for recoveries is greater than the allowable maximum) are noticed, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements, which may be compound specific. Check with the project manager about such requirements. Professional judgment may be used by the user (client) in evaluating sample data qualification options in conjunction with other QC data for the project.

11.10 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike recovery data are used to evaluate the accuracy of the analytical method on project samples. The evaluation is performed by comparing the %R for target analytes to the DoD QSM limits established for the LCS samples.

11.10.1 Document MS/MSD recoveries in instrument printouts and in LIMS generated reports as shown in Appendix 7. Flag QC outliers with "ME" and "ME*" on client reports. The MS/MSD percent recoveries sould be within QC limits (Tables 3 and 4). Perform one manual calculation using raw data and extraction sheet information.

11.10.2 MS data evaluation is more complex than method blank or LCS data evaluation since they are complicated by matrix effects in addition to sample preparation and analysis errors. The heterogeneity of grab soil samples, and potential non-representativeness of sequentially collected water samples, etc. further complicate the evaluation since it is assumed that the native concentrations are constant in triplicate analyses. In addition, concentrations of the target analytes in the sample can also far exceed spike amounts. If the native concentration of target analytes in the sample chosen for spiking is high relative to the spiking concentration, the differences in the native concentration between the unspiked sample and the spiked samples may also become very significant.

11.10.3 In addition to their reluctance to pay for MS and/or MSD analyses, most clients may not know what samples to collect for MS analysis. Clients may not collect sufficient samples, have no knowledge of the native analyte concentrations in the chosen sample, and may not have knowledge of MS spiking levels that are appropriate either for the sample or their project. For procedures with multiple target analytes, spiking levels may be inappropriate for one or more analytes, if samples that contain target analytes are chosen either in the field or by the laboratory. If no project samples are collected for MS analysis, samples from another project used for MS analysis may not be representative of the project samples. When project samples (e.g. surface soil) are chosen, their MS recovery characteristics may be different from those of other samples (e.g. sub-surface soil) in the same batch.

11.10.4 ACTION - If the sample chosen for MS/MSD is inappropriate, notify the client. When additional payments are authorized for re-analysis, re-extract and/or reanalyze the MS/MSD samples after employing cleanup

procedures (if applicable), dilution techniques to minimize matrix interference, or using other project samples that appear to be clean, when sufficient quantities are available. Re-spike the sample at a higher level (e.g., at approximately two to four times the sample concentration), then reanalyze the sample based on project-specific requirements. It is the responsibility of the client at all times to select appropriate samples for MS analysis, make payments for additional analyses, and to offer necessary guidance as needed to meet their data quality objectives.

11.10.5 ACTION - If the matrix effect cannot be resolved, discuss the situation in the case narrative. No action may be taken by the laboratory to qualify associated sample data based on MS/MSD recovery data alone. Professional judgment may be used by the user (client) in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

11.10.6 ACTION – No action is taken on MS/MSD recoveries data alone to qualify all associated samples. Since matrix interference could lead to lead to serious analytical problems, the supervisor should be consulted when significant outliers are noted. Document QC outliers for %recovery and RPD on the checklist and on client reports and draw attention to the QC outliers on the case narrative. No action may be taken on MS/MSD recovery data alone either to reanalyze samples and/or to qualify associated samples either by the analyst or by the end user. Professional judgment may be used by the user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

11.11 INTERNAL STANDARD PERFORMANCE – Internal Standard (IS) performance criteria ensures that the GC sensitivity and response is stable during each run.

11.11.1 IS area counts (use mean IS area from ICAL for the %D of IS area) must not vary by more than a factor of 2 (-50% or + 100%) from the associated ICAL/Calibration Verification standard. The internal standard areas for the CCV should not differ by a factor of 2 (-50% or + 100%) from the initial calibration of the same standard.

11.11.2 The retention time of the internal standard must not vary by more than 30 seconds from the associated daily calibration standard.

11.11.3 ACTION – If an internal standard is outside of QC limits of the associated daily standard, then re-analysis is required or a dilution may be required if it appears that interference's occur from target and non-target analytes at high concentrations relative to the ICAL. Do not dilute the sample to the point where all target analytes are non-detect. Professional judgement must be used when dilutions are required for IS criteria.

11.11.4 ACTION – If an internal standard is outside of QC limits of the associated ICAL or CCV standard, correct the problem and re-analyze samples with

a valid CCV. If re-analysis has been performed and it is still out, it is of the professional judgement of the analyst to dilute or not to dilute the sample further so as to not to dilute out any target analytes.

11.11.5 ACTION - Report results without qualification describing the QC problem in the case narrative. The user (client) may qualify positive results for compounds quantitated using the affected IS as estimated (J). If a very low IS area is reported, indicating a loss of sensitivity, non-detects may require qualification as invalid (I) or rejected (R). Document recommendation for the client in the case narrative.

11.11.6 ACTION – If an IS retention time varies by more than +/- 30 seconds, the total ion profile must be checked for any false positives or negatives. If a large shift has occurred, then professional judgement must be used as to whether reanalyze or not. See previous section for guidance.

11.12 TARGET COMPOUND IDENTIFICATION – Criteria for GC/ECD qualitative analysis is to minimize the number of erroneous positive or negative identifications of compounds.

11.12.1 Positive compounds must be within +/- 0.06 relative time (RRT) units of the standard RRT. The results should be >MDL or other threshold for reporting positives (e.g. LLR, RL) on both columns.

11.12.2 The RPD for sample amounts from the two columns for identified compounds should be within <40%. Document RPD calculations (See Appendix 6 for details) in Sample Result worksheets (Form-10 equivalents).

11.12.3 ACTION - If the RPD for positive results are <40%, report the result from the primary column without any flag.

11.12.4 ACTION - If the RPD is >40%, report the result from the primary column with a “J” flag. The result from the confirmation column may be reported with a “J” flag at the discretion of the analyst, if intererence is suspected for the compound of interest in the primary column.

11.12.5 ACTION – Professional judgement must be used when applying the criteria for GC/ECD analysis of target analytes. Take into account factors such as matrix effect and column bleed.

12.0 REFERENCES

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12.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8081B, Organochlorine Pesticides by Gas Chromatography, Draft Revision 2A, May 1999.

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12.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8082A, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Draft Revision 1A, May 1999.

12.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.

12.6 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

12.7 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3500B, Organic Extraction and Sample Preparation, December 1996.

12.8 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3520C, Continuous Liquid-Liquid Extraction, December 1996.

12.9 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3510C, Separatory Funnel Liquid-Liquid Extraction, December 1996.

12.10 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3511, Organic compounds in water by microextraction, Revision 0, November 2002.

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12.12 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3541, Automated Soxhlet Extraction, December 1996.

12.13 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3545, Presuurized Fluid Extraction, December 1996.

12.14 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3550B, Ultrasonic Extraction, December 1996.

12.15 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3570, Microscale Solvent Extraction (MSE), Revision 0, November 2002.

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12.17 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3610B, Alumina Cleanup, December 1996.

12.18 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3611B, Alumina Column Cleanup and Separation of Petroleum Wastes, December 1996.

12.19 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3620B, Florisil Cleanup, December 1996.
12.20 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3620C, Florisil Cleanup, Revision 3, November 2000.
12.21 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3630C, Silica Gel Cleanup, December 1996.
12.22 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3640A, Gel-Permeation Cleanup, December 1996.
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12.25 Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, EPA, 40 CFR Part 136, Method 608: Chlorinated Pesticides, October 1984.
12.26 Methods for the Determination of Organic Compounds in Drinking Water, Supplement II, EPA/600/4-88-039, Method 508, Determination of Chlorinated Pesticides in Water by Gas Chromatography with an Electron Capture Detector, July 1991.
12.27 Methods for the Determination of Organic Compounds in Drinking Water, Method 508.1, Determination of Chlorinated Pesticides in Water by Gas Chromatography with an Electron Capture Detector.
12.28 USEPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-media, Multi-concentration, OLM02.0.
12.29 USEPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-media, Multi-concentration, OLM03.0.
12.30 USEPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-media, Multi-concentration, OLM04.0.
12.31 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.
12.32 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.
12.33 AML Standard Operating Procedures, Q-Water-RC.
12.34 AML Standard Operating Procedures, Q-WasteMgmt-RD.
12.35 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
12.36 AML Standard Operating Procedures, Q-Controlcharts-RC.

Table 1

List of Default Target Compounds or Analytes for Organochlorine Pesticides Analysis (EPA Methods 508.1, 608, and 8081A)

Compound	CAS Registry No.
Aldrin	309-00-2
α-BHC	319-84-6
β-BHC	319-85-7
γ-BHC (Lindane)	58-89-9
δ-BHC	319-86-8
α-Chlordane	5103-71-9
γ-Chlordane	5103-74-2
Technical Chlordane	57-74-9
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan sulfate	1031-07-8
Endrin	72-20-8
Endrin aldehyde	7421-93-4
Endrin ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
Methoxychlor	72-43-5
Toxaphene	8001-35-2

Table 2

List of Optional Target Organochlorine Pesticide Analytes (EPA Methods 508.1, 608, and 8081A)

Compound	CAS Registry No.
Alachlor	15972-60-8
Captafol	2425-06-1
Chlorobenzilate	510-15-6
Chloroneb	2675-77-6
Chloropropylate	99516-95-7
Chlorothalonil	1897-45-6
DBCP*	96-12-8
DCPA	1861-32-1
Diallate*	2303-16-4
Dichlone	117-80-6
Dichloran	99-30-9
Dicofol	115-32-2
Etridiazole	2593-15-9
Halowax-1000	58718-66-4
Halowax-1001	58718-67-5
Halowax-1013	12616-35-2
Halowax-1014	12616-36-3
Halowax-1051	2234-13-1
Halowax-1099	39450-05-0
Hexachlorobenzene*	118-74-1
Hexachlorocyclopentadiene*	77-47-4
Isodrin*	465-73-6
Mirex	2385-85-5
Nitrofen	1836-75-5
PCNB	82-68-8
Permethrin	51877-74-8
Perthane	72-56-0
Propachlor	1918-16-7
Strobane	8001-50-1
trans-Nonachlor	39765-80-5
Trifluralin	1582-09-8

NOTE: Draft SW-846 Method 8081B contains the compounds marked with an asterisk superscript (*) in Table-1.

Table 3
Aqueous Sample MDL, LLR, MQL and DoD QSM Control and Marginal Exceedance (ME) Limits for LCS and MS Surrogate Recovery Limits

Chlorinated Pesticides Analysis													
Analytical Management Laboratories													
Preparation Methods:	3510C												
Analysis Methods:	8081A	8081B											
	MDL	**MDL**	**MDL**	**MDL**	**LLR**	**MQL**	**Ratio (RQ)**		**DoD QSM LCS & MS Control Limits**		**DoD QSM LCS & MS ME Limits**		**RPD for Dup.**
Compound	Spike	DB-5	DB-1701	Report	Report	Report	QL/DL	flag	Lower	Upper	Lower	Upper	Limit
	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L			**%R**	**%R**	**%R**	**%R**	%
α-BHC	0.025	0.0124	0.0087	**0.0124**	0.025	**0.10**	8.0		60	130	50	140	50
β-BHC	0.025	0.0221	0.0106	**0.0221**	0.025	**0.10**	4.5		65	125	55	135	50
γ-BHC	0.025	0.0154	0.0090	**0.0154**	0.025	**0.10**	6.5		25	135	10	155	50
δ-BHC	0.025	0.0036	0.0017	**0.0036**	0.025	**0.10**	27.9		45	135	30	150	50
Heptachlor	0.025	0.0176	0.0103	**0.0176**	0.025	**0.10**	5.7		40	130	30	145	50
Aldrin	0.025	0.0195	0.0096	**0.0195**	0.025	**0.10**	5.1		25	140	10	155	50
Heptachlor Epoxide	0.025	0.0210	0.0098	**0.0210**	0.025	**0.10**	4.8		60	130	50	140	50
γ-Chlordane	0.025	0.0201	0.0084	**0.0201**	0.025	**0.10**	5.0		60	125	50	135	50
Endosulfan I	0.025	0.0192	0.0090	**0.0192**	0.025	**0.10**	5.2		50	110	40	120	50
α-Chlordane	0.025	0.0202	0.0096	**0.0202**	0.025	**0.10**	5.0		65	125	55	135	50
Dieldrin	0.025	0.0163	0.0084	**0.0163**	0.025	**0.10**	6.2		60	130	50	140	50
DDE	0.025	0.0188	0.0072	**0.0188**	0.025	**0.10**	5.3		35	140	15	160	50
Endrin	0.025	0.0126	0.0186	**0.0186**	0.025	**0.10**	5.4		55	135	45	145	50
Endosulfan II	0.025	0.0154	0.0087	**0.0154**	0.025	**0.10**	6.5		30	130	10	150	50
Endrin Aldehyde	0.025	0.0123	0.0054	**0.0123**	0.025	**0.10**	8.1		55	135	40	150	50
DDD	0.025	0.0134	0.0103	**0.0134**	0.025	**0.10**	7.4		25	150	10	170	50
Endosulfan Sulfate	0.025	0.0117	0.0078	**0.0117**	0.025	**0.10**	8.5		55	135	40	150	50
DDT	0.025	0.0159	0.0095	**0.0159**	0.025	**0.10**	6.3		45	140	30	155	50
Endrin Ketone	0.025	0.0153	0.0082	**0.0153**	0.025	**0.10**	6.5		75	125	70	135	50
Methoxychlor	0.025	0.0258	0.0082	**0.0258**	0.025	**0.10**	3.9		55	150	40	165	50
Surrogates:													
DCB									30	135	30	135	
TCMX									25	140	25	140	
Criteria for the ratio (RD) of mean amount found to MDL: >1 but <5 and for MQL/MDL ratios (RQ) that are less than 3.													
QC limit for RPD is from the USACE document, Appendix I, EM 200-1-3, February 1, 2001.													
LCS (extended to the MS) %R limits are from the DoD QSM, Final Revision 2, June 2002.													
The following criteria will apply for marginal exceedances:													
Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).													
NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.													

Table 4
Soil Sample MDL, LLR, MQL and DoD QSM Control and Marginal Exceedance (ME) Limits for LCS and MS Surrogate Recovery Limits

Chlorinated Pesticides Analysis													
Analytical Management Laboratories													
Preparation Methods:	3550	3541											
Analysis Methods:	8081A	8081B											
	MDL	**MDL**	**MDL**	**MDL**	**LLR**	**MQL**	**Ratio (RQ)**		**DoD QSM LCS & MS Control Limits**		**DoD QSM LCS & MS ME Limits**		**RPD**
Compound	Spike	DB-5	DB-1701	Report	Report	Report	QL/DL	Flag	Lower	Upper	Lower	Upper	Limit
	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg			**%R**	**%R**	**%R**	**%R**	%
α-BHC	1.67	0.199	0.134	**0.199**	0.5	**1.67**	8.4		60	125	50	135	50
β-BHC	1.67	0.308	0.493	**0.493**	0.5	**1.67**	3.4		60	125	50	135	50
γ-BHC	1.67	0.227	0.253	**0.253**	0.5	**1.67**	6.6		60	125	50	135	50
δ-BHC	1.67	0.058	0.065	**0.065**	0.5	**1.67**	25.5		55	130	45	145	50
Heptachlor	1.67	0.228	0.327	**0.327**	0.5	**1.67**	5.1		50	140	35	155	50
Aldrin	1.67	0.306	0.424	**0.424**	0.5	**1.67**	3.9		45	140	30	155	50
Heptachlor Epoxide	1.67	0.328	0.481	**0.481**	0.5	**1.67**	3.5		65	130	55	140	50
γ-Chlordane	1.67	0.292	0.459	**0.459**	0.5	**1.67**	3.6		65	125	55	135	50
Endosulfan I	1.67	0.282	0.443	**0.443**	0.5	**1.67**	3.8		15	135	10	155	50
α-Chlordane	1.67	0.292	0.475	**0.475**	0.5	**1.67**	3.5		65	120	55	130	50
Dieldrin	1.67	0.241	0.408	**0.408**	0.5	**1.67**	4.1		65	125	55	135	50
DDE	1.67	0.281	0.406	**0.406**	0.5	**1.67**	4.1		70	125	60	135	50
Endrin	1.67	0.266	0.348	**0.348**	0.5	**1.67**	4.8		60	135	50	145	50
Endosulfan II	1.67	0.269	0.406	**0.406**	0.5	**1.67**	4.1		35	140	20	160	50
Endrin Aldehyde	1.67	0.299	0.464	**0.464**	0.5	**1.67**	3.6		35	145	20	165	50
DDD	1.67	0.338	0.389	**0.389**	0.5	**1.67**	4.3		30	135	10	155	50
Endosulfan Sulfate	1.67	0.175	0.293	**0.293**	0.5	**1.67**	5.7		60	135	50	145	50
DDT	1.67	0.260	0.268	**0.268**	0.5	**1.67**	6.2		45	140	30	155	50
Endrin Ketone	1.67	0.259	0.440	**0.440**	0.5	**1.67**	3.8		65	135	55	145	50
Methoxychlor	1.67	0.314	0.439	**0.439**	0.5	**1.67**	3.8		55	145	45	155	50
Surrogates:													
DCB									55	130	55	130	
TCMX									70	125	70	125	
Criteria for the ratio (RD) of mean amount found to MDL: >1 but <10 (for soil) and for MQL/MDL ratios (RQ) that are less than 3.													
QC limit for RPD is from the USACE document, Appendix I, EM 200-1-3, February 1, 2001.													
LCS (extended to the MS) %R limits are from the DoD QSM, Final Revision 2, June 2002.													
The following criteria will apply for marginal exceedances:													
Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).													
NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.													

Table 5
List of Appendices

Appendix Number	Appendix Topic
1	Target Compound List and their Concentrations In the Stock Standard
2	Retention Time Summary DDT and Endrin Breakdown Summary
3	Initial Calibration (ICAL) Summary (Form-6 equivalents) Instrument Printouts
4	Calibration Verification (ICV and CCV) Summary (Form-7 equivalents) Instrument Printouts and Standard Chromatograms
5	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
6	Sample Result Worksheets (Form-10 equivalents)
7	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
8	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
9	Surrogate Recovery Reports (Form-2 equivalents)
10	Method Detection Limit (MDL) Study Summary
11	QC Association Forms (Form-4 equivalents) Sample Preparation Logsheets Instrument Run Log
12	Sample Reports (Form-1 equivalents)
13	USACE Method Quality Objectives for Method 8081A AFCEE MQOs for Method 8081A DoD QSM MQOs and Limits for percent recoveries

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. O-OCPesticides-RD

ORGANOCHLORINE PESTICIDES BY GC/ECD (EPA Methods 508.1, 608 and SW846-8081B)

February 28, 2006

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP), which is based on EPA methods (508.1, 608, SW-846 8081A and 8081B-Draft Revision 2A) is used to determine the concentrations of various organochlorine pesticides in solid and liquid matrices, using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD). The compounds listed in Table 1 may be determined by either a single- or dual-column analysis system.

1.2 This SOP, which is based on EPA 508.1 and 608 and a revision of EPA SW-846 Method 8081 no longer includes Polychlorinated Biphenyls (PCBs) as Aroclors in the list of target analytes. The analysis of PCBs should be performed using AML SOP based on SW-846 Method Method 8082, which includes specific cleanup and quantitation procedures designed for PCBs analysis. Therefore, when performing analysis for 508.1 and 608 analytes, use AML SOP based on Method 8082 for PCBs analyses, and this SOP for the quantitative analysis of organochlorine pesticides and screening for PCBs.

1.3 If there is no information of the likely presence of PCBs, either employ a PCB-specific screening procedure such as an immunoassay (e.g., SW-846 Method 4020), or split the sample extract *prior to* any cleanup steps, and process part of the extract for organochlorine pesticide analysis and the other portion for PCB analysis using AML SOP based on Method 8082.

1.4 Surrogates are identical for the two methods and the sample preparation procedures are similar. Therefore, sample extracts and the method blank extract can be split prior to performing cleanup procedures that are specific for each analysis. Separate laboratory control samples and matrix spike samples should be analyzed for each method.

1.5 Several multi-component mixtures (i.e., Chlordane and Toxaphene) are listed as target analytes. When samples contain more than one multi-component analyte, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of multi-component analytes that have been subjected to environmental degradation or degradation by treatment technologies. These result in "weathered" multi-component mixtures that may have significant differences in peak patterns than those of standards.

1.6 This SOP describes anaylsis that employs a hardware configuration of two analytical columns joined to a single injection port. One injection is used for dual-column analysis.

1.7 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs (GC) and skilled in the interpretation of gas chromatogram.

1.8 This SOP may be used to analyze for additional compounds, which are shown in Table 2. The target list of compounds for specific projects may include one or more compounds from Table 2 in addition to the default list of compounds shown in Table 1.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample (approximately 1-L for liquids, 2-g to 30-g for solids) is extracted using appropriate matrix-specific sample extraction techniques.

2.2 Liquid samples are extracted at neutral pH with methylene chloride using procedures described in this SOP or AML SOPs based on SW-846 Method 3510C (separatory funnel), SW-846 Method 3520C (continuous liquid-liquid extractor), solid-phase extraction (Method 3535A) or other appropriate technique. The SW-846 methods including the overview (Method 3500B) should be used for additional guidance on cleanup procedures.

2.3 Solid samples are extracted with methylene chloride-acetone (50:50) using AML SOPs based on Method 3540C (Soxhlet), Method 3541 (automated Soxhlet), Method 3545 (pressurized fluid extraction), Method 3550B (ultrasonic extraction), microwave extraction (Method 3546) supercritical fluid extraction (Method 3562) or other appropriate technique. The SW-846 methods including the overview (Method 3500B) should be used for additional guidance on cleanup procedures.

2.4 This SOP is intended primarily for low-level definitive laboratory analysis of aqueous and soil samples for organochlorine pesticides using procedures shown in Sections 2.2 and 2.3. With appropriate modifications, microextraction procedures (SW-846 Methods 3511/3570) may also be used to support rapid turnaround analysis to support environmental remediation efforts. The microextraction procedures that are described in this SOP are similar to those described in SW-846 method 8011 and EPA Method 504.1.

2.5 A variety of cleanup steps may be applied to the extract, depending on the nature of the matrix interferences and the target analytes. Suggested cleanup procedures include AML SOPs based on SW-846 methods that employ alumina (Method 3610B and 3611B), Florisil (Method 3620B), silica gel (Method 3630C), gel permeation chromatography (Method 3640A), and sulfur (Method 3660B). The SW-846 methods including the overview (Method 3600C) should be used when a suitable AML SOP is unavailable during method development and/or implementation.

2.6 After cleanup, the extract is analyzed by injecting a 1-µL sample into a gas chromatograph with a narrow- or wide-bore fused silica capillary column and electron capture detector (GC/ECD).

3.0 INTERFERENCES

3.1 Interferences can be caused by a number of sources in the extraction and analyses stages. Quality control measures, such as the analysis of calibration blanks, method blanks, and solvent blanks can be used to monitor for and determine the interference.

3.2 Contamination may occur when a high-concentration and a low concentration sample are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. A high concentrated sample should be followed by the analysis of solvent to check for possible cross contamination.

3.3 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination should be expected with sediment samples. AML SOPs based on SW-846 Method 3660B is suggested for removal of sulfur. Since the recovery of Endrin aldehyde (using the TBA procedure) is drastically reduced, this compound must be determined prior to sulfur cleanup. The SW-846 methods should be used when a suitable AML SOP is unavailable during method development and/or implementation.

3.4 Halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. Certain co-eluting organophosphorus pesticides are eliminated by AML SOPs based on Method 3640A (gel permeation cleanup - pesticide option). Co-eluting chlorophenols may be eliminated by using Method 3630 C(silica gel), Method 3620B (florisil), or Method 3610B (alumina). Polychlorinated biphenyls (PCBs) also may interfere with the analysis of the organochlorine pesticides. The problem may be most severe for the analysis of multicomponent analytes such as Chlordane, Toxaphene, and Strobane. If PCBs are known or expected to occur in samples, the analyst should consult AML SOPs based on SW-846 Methods 3620B and 3630C for techniques that may be used to separate the pesticides from the PCBs. The SW-846 methods should be used when a suitable AML SOP is unavailable during method development and/or implementation.

3.5 The following compounds may coelute using the dual-column analysis scheme. In general, the DB-5 column resolves fewer compounds that the DB-1701.

DB-5	Permethrin/Heptachlor epoxide
	Endosulfan I/α-Chlordane
	Perthane/Endrin
	Endosulfan II/Chloropropylate/Chlorobenzilate
	4,4'-DDT/Endosulfan sulfate
	Methoxychlor/Dicofol
DB-1701	Chlorothalonil/β-BHC
	δ-BHC/DCPA/Permethrin
	α-Chlordane/*trans*-Nonachlor

Nitrofen, Dichlone, Carbophenothion, Dichloran exhibit extensive peak tailing on both columns. Simazine and Atrazine give poor responses on the ECD detector.

Triazine compounds should be analyzed using AML SOPs based on Method 8141 (NPD option) or the original SW-846 method.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph – Two Hewlett Packard (Agilent) 5890 Series II gas chromatographs (GCs) are used. The systems are complete with a temperature-programmable GC for splitless injection, all accessories, including syringes, analytical columns and gases, and Agilent 7673 or 7673A autosamplers. The column flow rate remains constant throughout temperature program operation.

4.2 Gas chromatographic columns – The instrument is operated in the dual column mode. The following GC capillary columns are employed:

4.2.1 A 30 meter x 0.32 mm ID capillary column coated with DB-5 (J&W Scientific) with a film thickness of 0.25 um.

4.2.2 A 30 meter x 0.32 mm ID capillary column coated with DB-1701 (J&W Scientific) with a film thickness of 0.25 um.

4.2.3 Other equivalent columns may be used in place of both columns.

4.3 Data system - Hewlett Packard's Environquant is used to allow continuous acquisition and storage on machine-readable media of all data obtained throughout the duration of the GC program.

4.4 Microsyringes - 10, 25, 100, 250, 500 and 1000 uLs.

4.5 Balance - Analytical, 0.0001 grams and top loading, 0.1 grams.

4.6 Disposable pipettes - Pasteur

4.7 Test tubes - 5 ml to 8 ml, with Teflon lined screw caps.

4.8 Separatory funnels - 2000 mL

4.9 Kuderna-Danish (K-D) apparatus a sdescribed below:

4.8.1 KD Tubes

4.8.2 KD Evaporation flask (500 mls), attached to concentrator tubes

4.8.3 Three ball macro snyder column

4.8.4 Clamps

4.10 Boiling Chips - solvent extracted

4.11 Heated water bath

4.12 Tekmar Sonic Disrupter

4.13 Glass wool

4.14 Glass beads, solvent-rinsed, bake in 400□C oven for approximately one hour

4.15 Stainless steel table spoons

4.16 IKA Automated Soxhlet Extractors – Six 4-position units equipped with heating devices, refrigerated circulating coolers, personal computer for instrument control, and micron filters to hold samples.

4.17 Soxtec Automated Soxhlet Extractors – Three 6-position units (HT-6) and two 2-position units, equipped with temperature-controlled oil bath (Soxtec). Accessories for the Soxtec equipment will include the following: Tecator bath oil (catalog number 1000-1886), contamination-free cellulose extraction thimbles (26 mm ID x 60 mm, catalog number 1522-0034), glass extraction cups (80 mL, catalog number 1000-

18200, thimble adapters (catalog number 1000-14660 and Viton seals (catalog number 1000-2516).

4.18 Circulating refrigerated coolers

4.19 Mechanical shakers

4.20 40-mL VOA vials (or equivalent) for microextraction of aqueous samples (SW-846 Method 3511) for rapid turnaround analysis (in support of remediation efforts)

4.21 Polytetrafluoroethylene (PTFE) tubes (25-mL capacity) for microscale solvent extraction (SW-846 Method 3570) of soil and waste samples for rapid turnaround analysis (in support of remediation efforts)

4.22 Mechanical centrifuge

4.23 Injector vials

4.24 Sonic Disrupter - Tekmar Model TM500 Sonic Disruptor with 500-watt power and pulsing capabilities. Use with ¾" horns and for guidance on use, see AML SOP on sonicator tuning and maintenance.

4.25 Tekmar Model V1A 3/4" Horn (probe).

5.0 REAGENTS

5.1 It is assumed that all inorganic/organic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where available. Any other reagents must demonstrate high purity without lessening the accuracy of the determination.

5.2 Organic free reagent water - All references to water in this method will be assumed to be organic free reagent water, except when referring to field samples.

5.3 Sulfuric acid solution

5.3.1 Slowly add 50 mL of sulfuric acid to 50 mL of water (1:1 v/v)

5.4 Solvents

5.4.1 Acetone – GC grade

5.4.2 Hexane – GC grade

5.4.3 Methylene chloride - GC grade

5.5 Stock Standard solutions - Available from Ultra Scientific (Catalog number PPM-808C). The list of analytes and concentrations are shown in in Appendix 1. Stock surrogate standards are also available from Ultra (catalog # ISM-320).

5.6 GC/ECD tuning standard solution. This solution, which is available from Supelco (Cat # 4-8397) contains DDT at 100ng/ml and Endrin at 50ng/ml. Prepare standard by diluting using 0.50ml of the commercial standard to 50ml of acetone.

5.7 Calibration Standards - Prepare a 100-ml stock standard solutions at 1000 ng/ml from Ultra Scientific catalog log number PPM-808C (20 compounds - conc. 1000ug/ml), & Ultra Scientific catalog number ISM-320 (TCMX & DCB 200 ug/ml) . Take 100ul of PPM-808C (Pesticides Compounds) & 0.500 of ISM-320 (TCMX & DCB) and dilute up to 100ml with acetone.

5.7.1 ICAL for Single Component Pesticides - The calibration curve consist of five points: 10ng/ml , 30ng/ml, 50ng/ml, 70ng/ml, & 100ng/ml. Preparation is as follows: 10ng/ml- (0.5ml of stock diluted to 50ml with acetone), 30ng/ml-(1.5ml of stock diluted to 50ml with acetone), 50ng/ml-(2.5ml of the stock diluted to

50ml with acetone), 70ng/ml-(3.5 ml of the stock diluted ot 50ml with acetone), 100ng/ml-(5.00ml of the stock diluted to 50ml with acetone).

5.7.2 CCV for Single Component Pesticides – Prepare a 50 ng/ml standard by diluting 2.5 ml of the stock to 50 ml with acetone

5.8 Sample surrogate spiking solution - The sample surrogate spiking solution is purchased as a shooter from Ultra-Scientific at 200 ng/ml cat. # ISM-321X. Spike 1.0 mL of the surrogate solution into each sample including QC samples. This solution contains decachlorobiphenyl and tetrachloro-m-xylene.

5.9 Matrix Spike (MS) spiking solution is prepared from a second source stock standard solution (Accustandard), which contains each target compound at a concentration of 1.0-mg/mL. Dilute 250-uL of thhis solution to 250-mL with acetone. The resulting matrix spike solution is at a concentration of 1000 ng/ml. Spike each MS and MSD sample with 0.250-ml of the 1000-ng/mL standard.

5.10 Laboratory Control Sample (LCS) spiking solution is the same as the matrix spike stnadard. Spike each sample with 0.25-mL of the 1000-ng/mL spiking solution.

5.11 Sodium Hydroxide Solution (10N) - Dissolve 40 grams of sodium hydroxide solution into 100 mLs of reagent water.

5.12 Solvents used in the extraction and cleanup procedures (AML SOPS based on appropriate 3500 and 3600 series methods) include n-hexane, diethyl ether, methylene chloride, acetone, ethyl acetate, and isooctane (2,2,4-trimethylpentane). The solvent must be exchanged to n-hexane or isooctane prior to analysis. Soil Sample should be extracted in a 50%Acetone/50 % Methylene Chloride solution**.**

5.13 Multicomponent pesticides - Separate calibration standards are required for each multi-component target analyte (e.g., Toxaphene and Chlordane). NOTE: The analysts should evaluate the Toxaphene standard carefully. Some Toxaphene components, particularly the more heavily chlorinated components, are subject to dechlorination reactions. As a result, standards from different vendors may exhibit marked differences which could lead to possible false negative results or to large differences in quantitative results.

5.13.1 Default ICAL Standard -

5.14 Internal standard. The compound, 1-bromo-2-nitrobenzene is used as an internal standard for the dual-column analysis. Prepare an intermediate solution of 1-bromo-2-nitrobenzene (50-ug/L) by diluting 1.0-mL of the commercial stock solution (Ultra, cat # PPS-351, 5,000-ug/mL) to 100-mL with acetone or iso-octane. Dilute 10-mL of this intermediate solution (50,000-ng/mL) to 100-mL with acetone to prepare the working standard at a concentration of 5,000-ng/mL. Spike 10 µL of this solution into each 1 mL of sample extract and calibration solutions. The final concentration of the internal standard in sample extracts and standards should be 50 ng/mL.

5.15 Sodium sulfate, granular, anhydrous (Na_2SO_4) for drying samples and sample extracts, ACS reagent grade.

6.0 SAMPLE PRESERVATION HANDLING & STORAGE

6.1 One litre amber glass bottles with Teflon lined screw caps may be used for collecting aqueous samples in accordance with EPA SW-846 method 8081A. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific,

etc.). Provide 2 bottles for each water sample to be collected. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.

6.2 One 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples in accordance with EPA SW-846 method 8081A. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each solid sample. Quality control samples such as MS and MSD should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. These samples may be shared with samples collected for other analyses such as semivolatiles (8270C), PCBs (8082), Diesel (DRO, 8015-mod.) and other pesticides (8140, etc.).

6.3 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Analytical data generated outside of the specified holding times must be considered to be minimum values only. Such data may be used to demonstrate that a waste is hazardous where it shows the concentration of a constituent to be above the regulatory threshold but cannot be used to demonstrate that a waste is not hazardous. The holding time for the extraction of aqueous samples, which includes TCLP leachates is 7 days from the date of sample collection or TCLP leachate preparation. The holding times for the extraction of soil, sediment and waste samples, which includes TCLP leachate preparation is 14 days from the date of sample collection. The holding time for the instrumental analysis of sample extracts is 40 days from the date of extraction.

7.0 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control contain a number of regulated compounds at high concentrations. The toxicity of these compounds present in such complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Aqueous and soil samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters after the solvent(s) have been allowed to evaporate in a hood.

9.0 SAMPLE EXTRACTION

9.1 Preparation of Sediment and Soil Samples

9.1.1 Sample Extraction by Sonication – SW-846 Method 3550

9.1.1.1 Weight out 30-g of the soil sample to three significant numbers (±0.1-g) into 500-mL beakers. Record the exact weight in the organic Extraction logbook (See Appendix 11 for documentation). Add enough sodium sulfate (60-g) to dry the soil and mix well with a spoon. When the soil is dry (freely flowing powder), add 1.0-mL of surrogate solution to all the samples (including QC) and 0.25-ml of the appropriate spiking solutions to the LCS/LCSD & MS/MSD.

9.1.1.2 Add 100-mL of the solvent mixture (methylene chloride:acetone 50:50 v/v) to the beaker and mix the contents with a spoon for 1 minute. Place the bottom surface of the ¾" horn about ½" below the surface of the solvent, but above the sediment layer. The horn should be properly tuned in accordance with the manufacturer instructions (see AML SOP on sonicator tuning). Extract ultrasonically for 3 minutes, with output control knob set at 10 (full power) and with mode switch on Pulse (pulsing energy rather than continuous energy) and percent-duty cycle knob set at 50% (energy on 50% of time and off 50% of time). Verify that the soil is free flowing and not in clumps and that the slurry has a smooth rolling action with each pulse of energy. Decant the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus.

9.1.1.3 Extract the sample twice more with 100-mLs of methylene chloride/acetone solvent mixture. The solution should be sonicated for 3 minutes each time. Decant the solution in to the same K-D as in the previous step.

9.1.1.4 Add two boiling chips to the K-D flask and attach a three-ball Snyder column. Prewet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1-ml, add 5-mL of hexane and continue with solvent removal. When the extract reaches 1 to 2-mL remove the K-D flask and allow to cool for at least 10 minutes.

9.1.1.5 Quantiatively transfer the extract to a 10-mL culture tube equipped with a PTFE-lined cap with graduation at 5-mL. Adjust the final volume to 5-mL with hexane. Proceed to analyze by GC/ECD if no cleanup procedures are required. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 50-ng/µL.

9.1.2 Sample Extraction by Automated Soxhlet Procedure A – SW-846 Method 3541

9.1.2.1 Automated Soxhlet Extraction Procedure-A involves IKA equipment, which are capable of handling 30-g sample aliquots. The set-up consists of six 4-position extractors to handle a total of 24 samples, which will include up to 5 QC samples (MB, LCS, LCSD, MS, and MSD). The six extractors, equipped with independent heating and cooling systems are controlled by a single personal computer. Follow manufacturer instructions for setting up and operating the IKA Soxhlet equipment.

9.1.2.2 Weight out 30-g of the soil sample to three significant numbers (±0.1-g) into 500-mL beakers. Record the exact weight in the organic Extraction logbook (See Appendix 11 for documentation). Add enough sodium sulfate (60-g) to dry the soil and mix well with a spoon. When the soil is dry (freely flowing powder), the sample is ready for Soxhlet extraction.

9.1.2.3 Clean and completely assemble the IKA equipment. Raise the condenser from the top vessel. Assemble the top vessel with the stainless steel filter base and a new disposable Teflon micron-sized filter paper. Transfer the soil/drier mixture quantitatively to the top vessel. Add 1.0-mL of surrogate solution to all the samples (including QC) and 0.25-mL of the appropriate spiking solutions to the LCS/LCSD & MS/MSD samples. Add 100-mL of the solvent mixture (methylene chloride:acetone 50:50) to the bottom vessel and tightly screw the top vessel on to the bottom vessel containing solvent. Replace the condenser unit on the top vessel. Turn the unit on and allow the unit to cycle (45-minutes). Completely drain the solvent from the sample chamber using the instrument cycle control mechanism. Repeat the extraction process twice using the same solvent contained in the bottom vessel. Decant the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus.

9.1.2.4 Add two boiling chips to the K-D flask and attach a three-ball Snyder column. Prewet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the

chamber should not flood. When the extract reaches approximately 1-ml, add 5-mL of hexane and continue with solvent removal. When the extract reaches 1 to 2-mL remove the K-D flask and allow to cool for at least 10 minutes.

9.1.2.5 Quantiatively transfer the extract to a 10-mL culture tube equipped with a PTFE-lined cap with graduation at 5-mL. Adjust the final volume to 5-mL with hexane. Proceed to analyze by GC/ECD if no cleanup procedures are required. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 50-ng/µL.

9.1.3 Sample Extraction by Automated Soxhlet Procedure B – SW-846 Method 3541

9.1.3.1 Automated Soxhlet Extraction Procedure-B involves Tecator Soxtec equipment, which are capable of handling only 10-g sample aliquots. The set-up consists of three 6-position extractors and two 2-position extractors to handle a total of 22 samples, which will include up to 5 QC samples (MB, LCS, LCSD, MS, and MSD). The five extractors are operated independent using three hot oil baths equipped with independent controllers. The condensers are cooled using a circulating refrigerant bath. Follow manufacturer instructions for setting up and operating the Soxtec equipment.

9.1.3.2 Weight out 10-g of the soil sample to three significant numbers (±0.1-g) into 500-mL beakers. Record the exact weight in the organic Extraction logbook (See Appendix 11 for documentation). Add enough sodium sulfate (10-g) to dry the soil and mix well with a spoon. When the soil is dry (freely flowing powder), transfer the entire contents of the beaker quantitatively to the rigid paper thimbles suspended from the stainless steel holders. Add 1.0-mL of surrogate solution to all the samples (including QC) and 0.25-mL of the appropriate spiking solutions to the LCS/LCSD & MS/MSD samples.

9.1.3.3 Clean and completely assemble the Soxtec extractors. Check the heating oil level in the automated Soxhlet unit and add oil if needed. See service manual for details. Set the temperature on the service unit at 80°C. Press the “MAINS” button and verify that the switch lamp is now in the “ON” position. Set the flow rate for the circulator at approximately 2-L/min to minimize solvent loss from the condensing unit.

9.1.3.4 Immediately transfer the thimbles containing the weighed samples into the condensers. Raise the knob to the "BOILING" position. The magnet will now fasten to the thimble. Lower the knob to the "RINSING" position. The thimble will now hang just below the condenser valve.

9.1.3.5 Insert the extraction cups containing boiling chips, and load each with 50-mL of extraction solvent. Using the cup holder, lower the locking handle, ensuring that the safety catch engages. The cups are now clamped into position. (The seals must be pre-rinsed or pre-extracted with extraction solvent prior to initial use.)

9.1.3.6 Move the extraction knobs to the "BOILING" position. The thimbles are now immersed in solvent. Set the timer for 60 minutes. The condenser valves must be in the "OPEN" position. Extract for the preset time.

9.1.3.7 After rinse time has elapsed, close the condenser valves by turning each a quarter-turn, clockwise. When all but 2- to 5-mL of solvent have been collected, open the system and remove the cups.

9.1.3.8 Transfer the contents of the cups to 15-mL graduated, conical-bottom glass tubes attached to the K-D flasks. Rinse the cups using methylene chloride and add the rinsates to the glass tubes.

9.1.3.9 Add two boiling chips to the K-D flask and attach a three-ball Snyder column. Pre-wet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1-ml, add 5-mL of hexane and continue with solvent removal. When the extract reaches 1 to 2-mL remove the K-D flask and allow to cool for at least 10 minutes.

9.1.3.10 Quantiatively transfer the extract to a 10-mL culture tube equipped with a PTFE-lined cap with graduation at 5-mL. Adjust the final volume to 5-mL with hexane. Proceed to analyze by GC/ECD if no cleanup procedures are required. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 50-ng/µL.

9.1.4 Microscale Solvent Extraction (SW-846 Method 3570) – This procedure should be used only in support of remediation efforts and for projects requiring relatively high detection limits. The Project Manager for specific projects may approve this extraction procedure.

9.1.4.1 Weight out 2.5-g (±0.5-g) of the soil sample to three significant numbers (±0.01-g) into 25-mL PTFE vials which has a PTFE screw cap. Record the exact weight in the organic Extraction logbook (See Appendix 11 for documentation). Add enough sodium sulfate (2.5-g) to dry the soil and 5 to 10 pre-cleaned glass beads for mixing the contents. Wipe the lips and threads of the tube with paper towels.

9.1.4.2 Add 1.0-mL of surrogate solution to all the samples (including QC) and 0.25-mL of the appropriate spiking solutions to the LCS/LCSD & MS/MSD samples. Add 12-mL of the extraction solvent to the tube and cap tightly. Shake the tubes vigorously until the slurry is free flowing. Use a mechanical shaker or vortex mixer, if necessary, breaking up any chunks with a metal spatula. Add more anhydrous sodium sulfate and manually mix as necessary to produce a free-flowing, finely divided slurry.

9.1.4.3 Extract the samples using a mechanical shaker or a rotating end-over-end unit for four hours. Decant or pipet the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus. Extract the soil sample in the PTFE tube twice with approximately 5-mL of the extraction solvent and shaking vigorously by hand for two minutes. Rinse the sodium sulfate with 2 to 3-mL of the extraction solvent.

9.1.4.4 Add two boiling chips to the K-D flask and attach a three-ball Snyder column. Pre-wet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1-ml, add 5-mL of hexane and continue with solvent removal. When the extract reaches 1 to 2-mL remove the K-D flask and allow to cool for at least 10 minutes.

9.1.4.5 Quantiatively transfer the extract to a 10-mL culture tube equipped with a PTFE-lined cap with graduation at 5-mL. Adjust the final volume to 5-mL with hexane. Proceed to analyze by GC/ECD if no cleanup procedures are required. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 50-ng/µL.

9.2 Preparation of Water Samples

9.2.1 Manual Liquid-Liquid Extraction (SW-846 Method 3510C)

9.2.1.1 Using a black marker, mark the meniscus of the water in the sample container. Pour the entire contents of the container into a 2-liter separatory funnel. Rinse the sample container with 3 aliquots of 20-mls methylene chloride and place the rinse in the separatory funnel. Carefully add tap water to the sample container up to the marked meniscus. Pour the distilled water into a 1-liter graduated cylinder. Record this measured volume in the organochlorine pesticides Extraction logbook (See Appendix 11 for documentation).

9.2.1.2 An alternate procedure, which may be employed to measure out water samples is described below. Using a 1-Liter graduated cylinder, measure out 1.00 liter ± 0.005 Liters (or lower volumes as low as 100-mL for high concentration samples) of the sample, and transfer it to a 2000-mL separatory funnel. Record the exact volume in the organic Extraction logbooks. This alternate procedure may be used to measure out lower sample volumes for the re-extraction of heavily contaminated samples with surrogate recoveries outside the QC limits, which are diluted out during analysis. This procedure may also be used to measure out smaller aliquots (e.g. 500-mL) of the sample for MS/MSD analysis, when additional sample containers were not collected for these analyses. NOTE: The measuring cylinder must be thoroughly cleaned using soap and water, rinsed with copius amounts of tap water, deionized water, and acetone prior to using it to measure out another environmental sample.

9.2.1.3 Using pH indicating paper (0-14 for general purpose and a narrow range paper 4 to 10 range for pH adjustment) record the pH of the sample (sample should be at a neutral pH). This is accomplished by dipping the tip of a Pasteur pipette into the water and placing the droplets of water on the pH paper. Never place the pH paper into the water sample. Adjust, if necessary, the pH to 7 using either 50/50 sulfuric acid solution or 10N sodium hydroxide solution. Add 1.0-mL of surrogate solution to all of the samples (including QC) and 0.25-mL of the appropriate spiking solution to the LCS/LCSD & MS/MSD.

9.2.1.4 Add 60-mLs of methylene chloride to the separatory funnel and shake for 2 minutes. Take care to vent the separatory funnel to relieve excess pressure. Allow the organic layer to separate from the water layer for a minimum of 10 minutes. Drain the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus. Repeat the extraction 2 more times (total 3 extractions) using 60-mLs of methylene chloride. Combine the extracts into a single KD vessel.

9.2.1.5 Add two boiling chips to the K-D flask and attach a three ball Snyder column. Prewet the Snyder column with 1 mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1-ml, add 5-mL of hexane and continue with solvent removal. When the extract reaches 1 to 2-mL remove the K-D flask and allow to cool for at least 10 minutes.

9.2.1.6 Quantiatively transfer the extract to a 10-mL culture tube equipped with a PTFE-lined cap with graduation at 5-mL. Adjust the final volume to 5-mL with hexane. Proceed to analyze by GC/ECD if no cleanup procedures are required. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 50-ng/µL.

9.2.2 Microextraction (SW-846 Method 3511) - This procedure should be used only in support of remediation efforts and for projects requiring relatively high detection limits. The Project Manager for specific projects may approve this extraction procedure. Samples may be collected in 40-mL VOA vials for this method if relatively volatile analytes are also included analysis using this determinative procedure. If they are collected in 1-L amber bottles, an aliquot may be transferred to a 40-mL VOA vial (including two additional aliquots for the sample selected for MS/MSD samples). Use reagent water contained in 40-mL vials for MB, LCS, and LCSD analyses. Use alternate pre-cleaned glass vials equipped with PTFE-lined caps, if the centrifuge does not accommodate 40-mL VOA vials.

9.2.2.1 Remove the cap from the VOA vial and use a disposable pipette to remove approximately 5-mL of water. Work with a single field or QC sample at a time and label the vial and the cap to minimize cross-contamination. Weigh the capped vial to the nearest 0.1-g and record it in the logbook. **NOTE:** Re-weigh the empty vial when the sample extraction procedure is completed (after completing Section 9.2.2.5) or weigh the vial before using it for microextraction. The difference between the two weights (g) should be used as sample volume (mL) assuming a density of 1.0 for aqueous samples.

9.2.2.2 Use a surrogate spiking solution (in acetone) that is 10-times more concentrated than those used for other extraction procedures (see Section 5.8) to maximize recoveries. Spike with 100-µL of the surrogate solution (10-times lower volume than for other extraction methods).

9.2.2.3 For spiked sample analysis, use LCS or MS spiking solutions (in acetone) that is 10-times more concentrated than those used for other extraction procedures (see Sections 5.9 and 5.10) to maximize recoveries. Spike with 25-µL of the LCS/MS spike solution (10-times lower volume than for other extraction methods).

9.2.2.4 Add exactly 2.0-mL (**NOTE:** This volume (2-mL) is the final extract volume for this procedure although half this volume is used for instrumental analysis) of methylene chloride using a gas-tight syringe or a class A volumetric pipette and approximately 12-g of anhydrous sodium chloride to the VOA vials. Replace the cap.

Shake the vials vigorously for five minutes or until the sodium chloride dissolves completely. The agitation may be performed using a mechanical shaker, if necessary. Briefly allow the phases to settle, then centrifuge at 500 times the gravity for 15-minutes.

9.2.2.5 Tilt the VOA vial to collect the lower methylene chloride to one section of the vial bottom. Pack a small pasteur pipette with glasswool and 100-mg of anhydrous sodium sulfate. Using a 1-mL gas-tight syringe, transfer approximately 1.5-mL of the lower methylene chloride layer through the pasteur pipette to a K-D tube. Add 1-mL of hexane through the pasteur pipette and concentrate using nitrogen blow-down to a small volume. When the extract reaches approximately 1-ml, add 1-mL of hexane and continue with solvent exchange.

9.2.2.6 Adjust the fnal volume to 1.0-mL of the dried extract in an injection vial. Proceed to analyze by GC/ECD if no cleanup procedures are required. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 50-ng/µL.

9.3 Sample Preparation for waste samples

9.3.1 If the waste is soluble in extraction solution, add approximately 1.0-g to a 15-mL culture tube. Record the exact weight in the Organic Extraction Log. Add 1.0-mL of the surrogate solution for samples and 1.0mL of surrogate plus 0.25-mL of appropriate spiking solution for LCS/LCSD & MS/MSD samples.

9.3.2 Dilute the extract to 10.0 mLs with extraction solution and proceed with GC/ECD analysis. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 50-ng/µL.

9.3.3 If the waste is not soluble in the extraction solution, the sample must be extracted like a sediment/soil sample using only 1.0-g instead of 30-g. Proceed to cleanup steps if the sample appears to be dirty.

9.4 Extract Cleanup Procedures

Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements. General guidance for sample extract cleanup is provided in this section, in AML SOPs based on SW-846 3600 Series methods or the SW-846 methods themselves. The SW-846 methods should be used when a suitable AML SOP is unavailable during method development and/or implementation.

9.4.1 The SOP based on SW-846 Method 3620 (florisil) is used as the deafult cleanup procedure by AML to separate organochlorine pesticides from aliphatic compounds, aromatics, and nitrogen-containing compounds.

Most if not all the samples and associated QC samples are subjected to this cleanup procedure prior to instrumental analysis.

9.4.2 If a sample is of biological origin, or contains high molecular weight materials, the use of SOPs based on Method 3640 (GPC cleanup - pesticide option) should be used. Frequently, one of the adsorption chromatographic cleanups (alumina, silica gel or Florisil) may also be required following the GPC cleanup.

9.4.3 AML SOPs based on SW-846 Method 3610 (alumina) may be used to remove phthalate esters.

9.4.4 AML SOPs based on SW-846 Method 3630 (silica gel) may be used to separate single component organochlorine pesticides from some interferants.

9.4.5 Elemental sulfur, which may be present in certain sediments and industrial wastes, interferes with the electron capture gas chromatography of certain pesticides. Sulfur should be removed by the technique described in AML SOPs based on SW-846 Method 3660.

9.4.6 Use the SW-846 method when a suitable AML SOP is unavailable during method development and/or implementation.

9.5 INSRUMENTAL ANALYSIS PROCEDURE

9.5.1 Dual-column analysis. The dual-column/dual-detector approach involves the use of two 30 m x 0.53 mm ID fused-silica open-tubular columns of different polarities, thus, different selectivities towards the target analytes. The columns are connected to an injection tee and separate electron capture detectors.

9.5.1.1 Retention times for the organochlorine analytes on the GC columns employed are shown in the instrument printouts, which are included in Appendix 2.

9.5.1.2 Intial temp – 80 degree

9.5.1.3 Hold time – 2.0 minute

9.5.1.4 3.0 degree/minute 250 degrees, hold for 2.0 minute.

9.5.1.5 Multi-component mixtures of Toxaphene and Chlordane are analyzed separately using the same conditions as above.

9.5.2 Tuning Solultion - DDT and Endrin are easily degraded in the injection port. Breakdown occurs when the injection port liner is contaminated high boiling residue from sample injection or when the injector contains metal fittings. Check for degradation problems by injecting a standard containing only 4,4'-DDT and Endrin. Presence of 4,4'-DDE, 4,4'-DDD, Endrin ketone or Endrin indicates breakdown. If degradation of either DDT or Endrin exceeds 15%, take corrective action before proceeding with calibration.

9.5.2.1 Calculate percent breakdown as follows:

$$\%\ \text{breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD + DDE)}}{\text{sum of all peak areas (DDT + DDE + DDD)}} \text{x}\,100$$

$$\%\ \text{breakdown of Endrin} = \frac{\text{sum of degradation peak areas (aldehyde + ketone)}}{\text{sum of all peak areas (Endrin + aldehyde + ketone)}} \text{x}\,100$$

9.5.2.2 The breakdown of DDT and Endrin should be measured before samples are analyzed. Injector maintenance and recalibration should be completed if the breakdown is greater than 15% for either compound. The calculation of DDT and Endrin breakdown is illustrated in Appendix 2.

9.5.3 Once the criteia shown above has passed, the initial calibration can be analyzed. Directly inject the initial calibration solution containing the single component pesticides in the following order: 10 STD, 30 STD, 50 STD, 70 STD, & 100 STD. Analyze a mid-level standard of the multi-component pesticides such as technical chlordane and toxaphene (or Strobane, if required) immediately after the analysis of a set of ICAL solutions.

9.5.4 The calibration curve for single component pesticides may be run from high to low instead of low too high as decribed. Summarize the initial calibration data in Form-5 equivalents (See Appendix 3). After the completion of the 50 STD standard ensure that all compounds have been appropriately identified. The analyst may choose to run a six point calibration, but it is not required by this method, but a five point is. One point may be dropped from the calibration if necessary, but not the low point, which is used to establish MQLs.

9.5.5 Tabulate the area response of the characteristic ions against concentration for each compound and each standard. Calculate response factors (RF) for each compound relative to the internal standards. For multi-component pesticides, use either the total area or 3 to 5 (4 to 6 for some programs) major peaks. The RF calculation is:

$$RF = (A_s C_{is}) / (A_{is} C_s)$$

where:

A_s = Area of the characteristic ion for the compound being measured
A_{is} = Area of the characteristic ion for the specific internal standard
C_{is} = Concentration of the specific internal standard
C_s = Concentration of the compound being measured

9.5.6 The average RF (RF_m) should be calculated and recorded for each compound using the five or six RF values calculated from the initial (5or 6-point) calibration curve. Using the RFs from the initial calibration, calculate and record the percent relative standard deviation (%RSD) and the mean response factor (RF_m) for each compound. The percent RSD is calculated as follows:

$$\% \text{ RSD} = ((SD) * 100\%/(RF_m)$$

where:

RF_m = mean of the 5 or 6 initial RFs for a compound
SD = standard deviation of the 5 or 6 initial RFs for a compound.

9.5.7 Linearity – If the % RSD of any single-component pesticide compound is 20% or less, then the relative response factor is assumed to be constant over the calibration range and the average relative response factor may be used for quantitation. It is desirable to review for trends as a function of the concentration for all compounds and exercise professional judgment to determine use of regression methods even for compounds that meet the <20% RSD criteria as described in the following section. Additionally, it is recommended that the fit of individual calibration points to the calibration line or curve be visually examined to determine if different calibration technique should be used. Since regression methods normally weigh more toward high-level standards, weighed regression methods are recommended if the concentration of concern is near low-level standards.

9.5.8 If the % RSD of any detected compound is greater then 20%, use least square regression methods to calculate sample concentrations. For regression methods, the instrument response is treated as the dependent variable (y) and the standard amount is the independent variable (x). When "Cs" is used as "x", use "As*Cis/Ais" as "y". When "Cs/Cis" is used as "x", use "As/Ais" as "y". Do not include origin (0,0) as an extra calibration point.

9.5.9 Although weighted least squares method is preferred for calculating concentrations more accurately at lower levels, commercially available software (check with the instrument manufacturer) should be used to perform regression procedures. A linear least squares equation ($y = a*x + b$) is preferred over non-linear calibration equations ($y = a*x^2 + b*x + c$). The coefficient of determination (COD or r^2) or correlation coefficient (r) should be used to measure the goodness of fit. For regression models to be used for quantitative purposes, the COD or r^2 must be greater than or equal to 0.99 and the correlation coefficient (r) should be greater than 0.995. A value of 1.00 for COD and/or "r" indicates a perfect fit.

9.5.10 Corrective action may be required if the criteria for %RSD, r, r2, or COD are not met.

9.5.11 An initial calibration (ICAL) should be considered a single event process. Consult the QA office or check applicable EPA methods (e.g. SW-846 Method 8000C and 8081A) for additional guidance on appropriate corrective action.

9.5.12 Re-fitting of ICAL – The selected model for ICAL may be subjected to an additional re-fitting check to establish representativeness of the model for each compound in each ICAL standard. This is an optional procedure, which is recommended but not required since automated data processing procedures may not be able to accommodate it. Calculate the %D between the calculated amount (Cc) for each compound in each ICAL standard from the ICAL model and its expected value (Ce). Ideally, the %D ((Cc-Ce) * 100 /Ce) for each compound in ICAL standard should meet the QC criteria for %RSD. It is possible for one or more ICAL standards to have %D exceeding the %RSD criteria, while the %RSD for ICAL model is within QC limits. The re-fitting procedure may be used to identify ICAL standards that need re-analysis or dropped from the calculation of ICAL model when re-analysis was not performed.

9.5.13 Check and optimize instrument operating conditions including automated peak integration procedures for appropriateness in addressing the chromatographic needs of the specific compound with problems.

9.5.14 If the problem appears to be associated with just one of the ICAL standards, that one standard may be reanalyzed once immediately (must be within 8-hours) after the ICAL. Discard all the results from the original analysis of the standard in question and replace it with the results from re-analysis.

9.5.15 When additional calibration standards (six or seven instead of five) are analyzed, narrowing the range of concentrations to be used for determining the goodness of fit is an acceptable procedure. Five or more standards should remain for the calculation of RSD or linear regression for each compound after one or more points are removed from the ICAL. More points (6 for second order and 7 for third order) may be needed for non-linear regression. It is generally prohibited to remove data points from within a calibration range, while still retaining the extreme ends of the calibration range. Do not discard a middle point unless it appears to have an obvious problem and six or more standards were analyzed for ICAL and re-analysis of the affected standard was not performed within the required time limits. Changes to the upper end of the calibration range will affect the need to dilute samples above the range. A change to the lower range is not recommended since it will affect the method quantitation limit (MQL) used to report data. The default MQLs, which are based on the lowest ICAL concentration are stored in the LIMS and project-specific changes are not practical.

9.5.16 The refitting procedures may lead to failed %Ds for upper level calibration standards for average response factor methods. The analyst may take alternate corrective actions such as the analysis of sample extracts under dilution for the affected compounds.

9.5.17 The refitting procedures may lead to failed %Ds for lower level calibration standards for regression methods. The analyst may take appropriate corrective action such as flagging the sample result as an estimated amount ("J" flag) at the lower level encountered in the sample.

9.5.18 Grand mean RSD – Previous versions of the EPA method(s) had provisions for the calculation of the grand mean %RSD for all target compounds in the ICAL solution, when the % RSD of any detected compound is greater than 20%. The grand mean %RSD should be <20% for ICAL to be acceptable. This procedure is discontinued. Criteria for Initial calibration are compound-specific.

9.5.19 The term "calibration verification" (CALV) is used to describe initial verification (ICV) of the initial calibration (ICAL) using second-source standards and subsequent continuing verifications (CCVs) using same-source standards. The response factors calculated during CALV are not used for sample quantitation since CALV standards are not used as "continuing calibration" standards permitted in some of the EPA methods.

9.5.20 Initial Calibration Verification (ICV) - Analysis of a second source ICV standard is required before any samples can be analyzed for USACE projects. Since second source standards are not employed for the analysis of LCS/LCSD and MS/MSD samples, ICV analysis is applicable for all projects. Calculate the percent difference (%D) or %Drift for the observed response factor in ICV from the mean RF as described for CCV. The %D or %drift should not exceed ±15% for each target compound. Document calibration verification in instrument printouts as shown in Appendix 2. Check the ICAL and ICV standards if it is not achievable for all the target compounds without any exception. Use professional judgment for non-USACE projects and for compounds that are not contaminants of concern (COC) for USACE projects. The ICV may be used as the beginning CCV for sample analysis.

9.5.21 If the above criteria are met, the analysis for analytical samples can begin.

9.5.22 Continuing Calibration Verification (CCV) – The initial calibration curve for each compound of interest must be checked and verified once every 12-hours with a calibration verification standard. Analyze the mid level STD and calculate the percent difference (%D) of the RRF from the average RRF for each compound. The %D for each compound should be <15%. If the %D for any compound is greater than 15%, the GC should be restored to proper operating condition before re-analyzing the CCV for all the compounds. A new ICAL should be genearted if the CCV does not meet QC criteria.

9.5.23 Grand mean %D – Previous versions of the EPA method(s) had provisions for the calculation of the grand mean %D for all target compounds in the CCV stanadard, when the %D of any detected compound is greater than 15%. The grand mean %D should be <15% for CCV to be acceptable. This procedure is discontinued. Criteria for CCV is compound-specific. Summarizes CCV in Form-7 equivalents (See Appendix 4).

9.5.24 Calibration Factors are used when external standards are used for calibration. This procedure is not used at AML and they are described only when they are necessary. Situations include internal standard coeluting with one of the traget compounds in one or more columns. When external standard calibration is employed, calculate the calibration factor for each analyte at each concentration, the mean calibration factor, and the relative standard deviation (RSD) of the calibration factors, using the formulae shown below. If internal standard calibration is employed, refer to Method 8000 for the calculation of response factors. Calculate the calibration factor for each analyte at each concentration as:

$$CF = \frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

Calculate the mean calibration factor for each analyte as:

$$\text{mean CF} = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

where n is the number of standards analyzed. Calculate the standard deviation (SD) and the RSD of the calibration factors for each analyte as:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF})^2}{n-1}} \qquad RSD = \frac{SD}{\overline{CF}} \times 100$$

If the RSD for each analyte is 20%, then the response of the instrument is considered linear and the mean calibration factor can be used to quantitate sample results. If the RSD is greater than 20%, then linearity through the origin cannot be assumed. The analyst must use a calibration curve or a non-linear calibration model (e.g., a polynomial equation) for quantitation. See SW-846 Method 8000C or AML SOPs (if available) based on this SW-846 Method for information on non-linear calibrations.

9.5.25 ANALYSIS OF SAMPLE EXTRACTS

9.5.25.1 Once the samples have been extracted and all system performance checks have been established as stated within this SOP, sample extracts can be analyzed. A 1-mL aliquot of the sample extract is spiked with 10-µLs of internal standard solution (1-bromo-2-nitrobenzene at 5000 ng/µL). The final concentration of internal standard is 50 ug/mL for each 1 mL extract.

9.5.25.2 If the response for any compound exceeds the initial calibration curve range of the GC/ECD system, extract dilutions must take place. Additional internal standard must be added to the diluted extract to maintain the required 50 ng/uL of each internal standard in the extract used for instrumental analysis. The diluted extract must be reanalyzed.

9.5.26 DATA INTERPRETATION

9.5.26.1 The qualitative identification of compounds determined by this method is based on retention time, and % difference between positive results on the two columns. The qualitative identification of multi-component pesticides such as chlordane, toxaphene or strobane is made from their pattern and from the presence of characteristic peaks. Technical BHC and Technical DDT are not identified by their pattern. They are identified by their individual components (α-BHC, δ-BHC, DDD, DDE, etc.). Compounds should be identified as present when the criteria below are met.

9.5.26.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component in both GC columns. The calculated amount should be >MDL (or >LLR or >RL when these are used to define the lowest level for reporting positives) on both columns.

9.5.26.3 The relative percent difference (RPD) between the results obtained from the two columns should be <40%. If RPD is >40%, the result(s) should be reported as an estimated quantity with a flag (e.g. "J"). If one column result is significantly higher (e.g., >40%), check the chromatogram to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baselines for both columns and baseline parameters established for peak integrations. A rising baseline may cause a lower result. Manual integration may be necessary in some cases to obtain accurate peak areas. If no peak integration anomalies are noted, check chromatographic conditions and the chromatograms for LCS and LCSD samples for good separation of target analytes and reproducibility of results from the two chromatographic columns. If no anomalies are noted, matrix interference and/or the presence of non-target compounds may be inferred. Report result(s) in accordance with guidance provided in Section 9.5.26.4

9.5.26.4 Reporting of the results should follow method guidance and guidance from regulatory agencies (federal, state, local) and guidance from clients (contract requirements, QAPP requirements, etc.). Client requirements, when available should address regulatory requirements. AML guidance, which is based on SW-846 Method 8000C will serve as the deafult guidance as described below:

9.5.26.4.1 Default AML Procedure: Report the lower of the two results from the primary and confirmation columns. Report results with RPD >40% as estimated ("J" flag).

9.5.26.4.2 Client guidance and analyst discretion are very important. Some clients may require one of the columns to be designated as primary and the second column as confirmatory. In such cases, results may need to be reported from the primary column unless interference is suspected for a specific compound in the primary column, which are not present in the confirmation column.

9.5.26.4.3 The SW-846 Method 8000 guidance has not been consistent over time. Although the most recent Method 8000 revision (Rev. C) recommends the reporting of the lower result, earlier revisions (e.g. Rev. B) required the reporting of the higher result since it was considered to be more protective of the environment. Some EPA regional offices may have issued memoranda, which may require the reporting of the higher result.

9.5.26.4.4 Some projects may require the reporting of results from both columns.

9.5.26.5 Confirmation of positives may also be made from GC/MS analysis of the pesticide extracts when applicable and from the identification of the pesticide compound from BNA (SVOCs) fraction, if analyzed. Document sample result in worksheets that are Form-10 equivalents (See Appendix 6)

9.5.27 QUANTITATIVE ANALYSIS

9.5.27.1 The internal standard technique is used to determine the concentration of an identified target compound. For water samples the calculation is as follows:

$$\text{Concentration } (\mu g/L) = \frac{(A_s) * (IS) * (V_e)}{(A_{is})\,(RF_m)\,(V_s)}$$

where:

A_s = Area for compound being measured.
IS = Concentration of the internal standard in the extract injected (µg/mL)
Ais = Area for the internal standard
RF_m = Mean Response factor for compound being measured
V_s = Volume of water (L) extracted
V_e = Volume of the final extract (mL), taking into account any dilutions made

9.5.27.2 For soil samples the calculation is as follows:

$$\text{Concentration } (\mu g/kg) = \frac{(A_s) * (IS) * (V_e) * (100)}{}$$

$$(A_{is}) * (RF_m) * (W_s) * (\%S)$$

where:

A_s = Area for compound being measured.
IS = Concentration of the internal standard in the extract injected (µg/mL)
Ais = Area for the internal standard
RF_m = Mean Response factor for compound being measured
W_s = Wet weight of soil sample (kg) extracted
V_e = Volume of the final extract (mL), taking into account any dilutions made

10.0 QUALITY CONTROL

10.1 Using this SOP will involve implementing the referenced EPA method at the laboratory. Method implementation involves the following: chromatographic separation of the target analytes on two GC columns, determination of retention time windows for each compound, determination of method detection limits for each sample matrix and/or preparation method, spiked sample analysis, initial demonstration of acceptable recoveries, etc. Method implementation procedures are described on the AML SOP on MDL.

10.2 Analytical data, which includes calibration data, sample preparation logs, sample data and quality control data should be gathered prior to review. Review checklist (See Appendix 5) should be used to document verification of calculations and adherence to method procedures. Exceptions should be documented. Samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on these checklists.

10.3 Summarize sample results using Form-1 Equivalents (See Appendix 12). Summarize QC results and QC recoveries in Form-1 and Form-3 equivalents respectively for MS/MSD (Appendix 7) and LCS/LCSD (Appendix 8).

10.4 Surrogate recovery should be within QC limits (DoD QSM, Revision 2, June 2002) for samples, spikes and blanks. Document surrogate recoveries in Form-2 equivalents (See Appendix 9) Flag recoveries that are outside the QC limits with an asterisk (*). If re-extraction and/or reanalysis has been performed, and the surrogate recoveries for reanalysis are within QC limits, report data based on acceptable surrogate recoveries. If the surrogate recoveries are outside the QC limits for the reanalysis, report data for both analyses with surrogate recovery flags.

10.5 Method blanks (MB) should be free of target compounds. The default QC limit for method blank is one half of the applicable MQL. See Tables 3 and 4 for a list of MDLs, LLRs, and MQLs. Details of the MDL determinations are included in Appendix 10. When project specific reporting limits are available, which may be greater than MQL, the project specific QC limit for MB is one half of the reporting limit. Samples associated with the method blank containing target compounds at levels greater than the QC limit will require re-extraction and reanalysis unless other criteria accepable to the client are used. Since method blanks may be contaminated when they are associated with samples containing high levels of target compounds, professional judgment must be exercised to determine samples requiring re-extraction and reanalysis.

10.6 LCS recoveries should be within QC limits (statistical control limits and DoD QSM limits) for the target compounds. Flag recoveries that are outside the quality control limits with “ME” and compounds outside the ME limits with “ME*”. See Tables 3 and 4 for QC limits. Contact supervisor for appropriate corrective action if more than the allowed number of compounds are outside the control limits or the marginal exccedance (ME) limits. Although data qualification may be sufficient for commercial clients, re-extraction, and/or reanalysis of associated samples will be

necessary for most DoD projects. Other constraints may include project schedule, turnaround time for re-analysis and the availability of additional sample volume.

10.7 MS recoveries should be within QC limits (DoD QSM) for the target compounds. Flag recoveries that are outside the control limits with "ME" and compounds outside the ME limits with "ME*". See Tables 3 and 4 for QC limits. Contact supervisor for appropriate corrective action if more than the allowed number of the compounds are outside the control limits or the marginal exccedance (ME) limits. Data qualification, re-extraction, and/or reanalysis of associated samples may be necessary depending on project requirements, turnaround time and other constraints.

10.8 Report sample results on Form-1 equivalents (See Appendix 12). Report target compounds that are present at levels greater than the reporting limit (RL) using "J" qualifiers if the levels are less than MQL. Compounds that were also detected in the associated MB should also be qualified with a "B" code. If the concentration of any of the target compounds exceed the upper limit of calibration (ICAL), flag the affected compounds data with an estimated or "E" flag. The extracts may need to be diluted and reanalyzed until the concentrations are within the calibration limits for normal turnaround analyses. For rapid turnaround analyses sample results may be reported with "E" qualifiers if sufficient time is not available for reanalysis.

11.0 **DATA VERIFICATION AND VALIDATION -** This section will provide guidance and procedures to assess if data produced using this SOP is valid and legally defensible. Document review and comments on the checklist for the raw data package or other detailedchecklists (See Appendix 5). Verify that sample preparation logs are complete and the instrument run logs are available for review (See Appendix 11). Anomalies and exceptions should be documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on the checklists. Army (USACE), Air Force (AFCEE) or Department of Defense (DoD) projects may have special requirements (See Appendix 13) that will require extra attention. A simple, universal procedure for evaluating and choosing appropriate corrective action doe not exist to deal with situations when one or more QC parameters are outside the acceptance window. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints.

11.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) projects may have additional requirements that are related to USACE laboratory validation (CX), USACE District requirements, government contract requirements, specific USACE project requirements or USACE Architect/Engineer (A/E) requirements. USACE-CX requirements (Summary of Method Quality Objectives for Method 8081A) and DoD requirements are included in Appendix 13.

11.1.1 The USACE requirements shown in Appendix 8 and elsewhere in this SOP are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the

legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may or may not be attainable for routine sample analysis. Since USACE MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

11.1.2 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality objectives and/or other QC requirements. Exceptions and data qualifications necessary should be documented on the checklists and eventually on case narratives.

11.1.3 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist, perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete. For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete. For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director will review the checklists (initial/date as necessary), prepare case narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

11.1.4 The blank criteria is one half of the applicable MQL (default criteria) or one half of the project specific reporting limit (which may be higher than MQL), whichever is applicable.

11.2HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

11.2.1 WATER - The holding times for semi-volatiles is 7 days from sampling.

11.2.2 SOIL – A 14 day holding is used for soil and waste samples.

11.2.3 TCLP – TCLP leachates must be extracted within 7 days after the TCLP extraction.

11.2.4 CHECK – Establish holding times by comparing the chain of custody sampling date with the date of the injection after purging. TCLP holding

times must be established by comparing first the COC to the leaching date and then the leaching date to the sample extraction date, followed by the extract injection date.

11.2.5 ACTION – If holding times have been exceeded, contact the client. All positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or innvalid (I).

11.3COLUMN PERFORMANCE – Column performance criteria for GC\ECD should meet criteria established in this SOP. Column performance is measured from the DDT breakdown to DDD and DDE and the endrin breakdown to its aldehyde and ketone (See Appendix 2 for details). Such breakdown should not exceed 15%. The breakdown should be checked every 12 hours.

11.3.1 Check to see if the column performance report is present and that it meet criteria above.

11.3.2 ACTION – If the breakdown is outside criteria, perform appropriate corrective action as described elsewher in this SOP and in the referenced SW-846 method, recalibrate and rerun the breakdown standard for an acceptable report. The calibration can not be run until the breakdown is in control. If full documentation on DDT/Endrin brekdown standard is not present, the package is considered incomplete.

11.4INITIAL CALIBRATION (**ICAL**)– Instrument calibration (See Appendix 3 for details) criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the GC/ECD is capable of producing data within a range that is linear. The ICAL includes initial verrification (ICV) of the ICAL with a second source standard for USACE and AFCEE projects (See Appendix 13 for details).

11.4.1 CHECK – Check to see if any other compounds are outside the criteria of 20% or less for %RSD. If a compound is outside the 20% criteria, check to see if regression methods are used. The goodness of fit should be measured with the coefficient of determination (COD) or related quantities (r^2 and r) greater than 0.99.

11.4.2 ACTION – Check and correct the standards, including the range of concantrations for appropriateness and repeat the ICAL analysis.

11.4.3 ACTION – Use a lesser number of peaks (minimum 3) if the criteria are not met for five peaks (desirable) selected for each multi-component analyte.

11.4.4 ACTION - Report the problem to the Project Manager and report detects and non-detects with aapropriate qualifiers for rapid TAT projects. Mention the ICAL anomaly in the case narrative.

11.5 INITIAL CALIBRATION VERIFICATION – Calibration verification includes initial calibration verification (ICV) and continuing calibration verifications (CCVs). A second source ICV is used to check the integrity of the ICAL standards used for calibrating the instrument. The 12-hour analytical shift begins with the injection of the ICV (instead of a CCV) for sample analyses that are performed immediately after an ICAL.

11.5.1 Target Compounds - Check to see if %D for any of the target compounds is outside the USACE QC criteria (<±15%). Check also for systematic errors that may result from bias between the two standards.

11.5.2 ACTION – When the problem is confined to a few specific target compounds, check peak separation, peak integration and integration parameters for anomalies. If no anomalies are found, check the ICAL and ICV standards for integrity. Replace one or more of the working standards (ICV first and ICAL later) for trouble shooting. Correct the problem(s) and repeat ICV analysis. Use professional judgment in selecting an appropriate corrective action for all projects.

11.5.3 ACTION - When a CCV immediately following the ICV is acceptable, peak integration and chromatograhic anomalies are not likely to be present. The analysis of standards from a third source may be necessary, if the inspection and replacement of ICV and ICAL standards fail to resolve the anomaly between ICAL and the second source ICV.

11.6 CONTINUING CALIBRATION VERIFICATION – The continuing calibration verification (See Appendix 4 for details) is used to check that the instrument is still operating under the linear range of the initial calibration and has remained stable every 12 hours.

11.6.1 IS area counts (use mean IS area from ICAL for the %D of IS area) must not vary by more than a factor of 2 (-50% or + 100%) from the associated ICAL. Check to see if the %D for any compounds are outside the criteria of 15% or less.

11.6.2 %D for Compounds present in the sample – The %D for the CCVs before and after the sample analysis should be within QC limits when internal standards are not used. The %D for the CCV before sample analysis should be within QC limits when internal stanadrds are used.

11.6.3 %D for Compounds not present in the sample – The %D for the CCVs before and after the sample analysis may be >(+15%). Corrective action is not required unless the %D is < (-15%).

11.6.4 ACTION – Corrective action must be performed for positive results with %D outliers. In exceptional cases (e.g rapid TAT analysis for screening level data), positive results for compounds not meeting the criteria may be qualified as estimated (J) by the user (client).

11.6.5 ACTION - Report non-detects without qualifiers and include a description of the anomaly in the case narrative.

11.6.6 ACTION – Perform more frequent CCVs (e.g every 10 samples) than required (every 12-hours) to avoid extensive re-analysis.

11.7METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Sample data qualification to alert blank contamination is performed by the laboratory. Sample data qualification based on all the blank results (laboratory MB, equipment blank, and other field blanks) is performed by the user (client). The processing of blanks helps eliminate reporting of false positives in project reports.

11.7.1 Method blanks should be free of all target anaytes and other interfering non-target analytes. To be acceptable, blank results should not exceed the QC limit, which is one half of the applicable MQL for any compound (default) or one half of the reporting limit (project specific).

11.7.2 Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples. If levels greater than the QC limit are present, inspect and correct the problem. Reanalyze all associated samples. Contact client if other criteria are applicable. The only exception may be quick turn around projects.

11.7.3 ACTION – All positive results are reported with a “B” code, if contaminants are found in the method blank.

11.7.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, then no action is taken.

11.7.5 ACTION – Any compound detected in the sample which were also detected in associated method blank at levels greater than the QC limit should be re-extracted unless other client criteria are applicable.

11.7.6 ACTION – If contaminants resulting in GC/ECD peak saturation are present, all affected compounds are qualified as invalid (I).

11.8SURROGATE RECOVERIES – Surrogates or system monitoring compounds are different structurally, but similar chemically to classes of target compounds. These surrogate compounds are added just prior to extraction of the samples and the percent recoveries of the surrogates are calculated and evaluated.

11.8.1 Verify that the surrogate recoveries (See Appendix 9 for details) for the surrogate compounds, DCB and TCMX are within the QC limits (laboratory control limits and DoD QSM control limits):

11.8.1.1 For aqueous samples, the DCB recoveries should be within the range, 30% to 135%. The TCMX recoveries should be within the range, 25% to 140%. Flag outliers with an asterisk (*).

11.8.1.2 For soil samples and other solid matrices, the DCB recoveries should be within the range, 55% to 130%. The TCMX

recoveries should be within the range, 70% to 125%. Flag outliers with an asterisk (*).

11.8.2 ACTION – When surrogate recoveries are outside control limits the sample must be re-analyzed. Reanalysis with or without cleanup may be performed under dilution, if necessary if original analysis should indicate high levels of target and non-target analytes.

11.8.3 ACTION - Reextraction and cleanup will be necessary if reanalysis does not resolve the problem. If surrogate recoveries are still outside the limits, report both sets of data and describe the problem in the case narrative.

11.9 LABORATORY CONTROL STANDARD (LCS) – The analysis of LCS samples is used to evaluate method competence in the absence of matrix effects. The LCS is evaluated by comparing the percent recovery for all of the target analytes to the laboratory QC limits, which are identical to the 3-sigma DoD QSM QC limits. See Appendix 10 for guidance on documenting LCS and LCSD concentrations and %R in QC summary report forms. The DoD QSM specifies the number of compounds that can exceed the 3-sigma QC limits based on statistical considerations. The LCS recovery for such compounds should not exceed the marginal exceedance 4-sigma (ME) limits. Evaluation procedures may include comparison of LCS recoveries with those determined by project-specific DQOs (See project QAPP, if available), laboratory statistical control limits, or USEPA guidance. Initially, the effect of QC failure on the samples should be evaluated. Regardless of this assessment, steps shall be taken to find the source of the problem and correct it.

11.9.1 CHECK - LCS and LCSD recoveries (See Appendix 8 for details) should be within QC limits (See Tables 3 and 4). Compare the %R results for each compound in LCS with laboratory QC limits to see if all the results are within the acceptable range. Flag outliers with “ME” if it is within the 3-sigma limits. Flag it with “ME*” if it is outside the 4-sigma ME limits.

11.9.2 CHECK - Compare observed %R results for each compound with the laboratory limits for LCS/LCSD recoveries (e.g. USACE criteria). Perform manual calculation for %R, if necessary for QC outliers. Document the number of compounds that exceeded the QC limit and compare it against the maximum recommended by DoD QSM. Document this review in checklists (Appendix 7) and in case narratives.

11.9.3 ACTION - Typically, the LCS would be reanalyzed for the failed analytes (ME*) only. If the second analysis fails, then the LCS, method blank, and all associated samples of the batch would be reanalyzed for the failed analytes only. If sufficient sample is not available for reanalysis, or if the corrective action is ineffective, discuss the situation with the client project manager (or chemist). Include a discussion of the situation in the case narrative. The case narrative should discuss the corrective action taken and any other information.

11.9.4 ACTION - For non-USACE projects, no action may need to be taken on LCS and/or LCSD recovery data alone either to reanalyze samples and/or to qualify associated samples. Providing data of known quality is

sufficient for most projects. If significant deviations (e.g. number of target compound exceeding QC limits for recoveries is greater than the allowable maximum) are noticed, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements, which may be compound specific. Check with the project manager about such requirements. Professional judgment may be used by the user (client) in evaluating sample data qualification options in conjunction with other QC data for the project.

11.10 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike recovery data are used to evaluate the accuracy of the analytical method on project samples. The evaluation is performed by comparing the %R for target analytes to the DoD QSM limits established for the LCS samples.

11.10.1 Document MS/MSD recoveries in instrument printouts and in LIMS generated reports as shown in Appendix 7. Flag QC outliers with "ME" and "ME*" on client reports. The MS/MSD percent recoveries sould be within QC limits (Tables 3 and 4). Perform one manual calculation using raw data and extraction sheet information.

11.10.2 MS data evaluation is more complex than method blank or LCS data evaluation since they are complicated by matrix effects in addition to sample preparation and analysis errors. The heterogeneity of grab soil samples, and potential non-representativeness of sequentially collected water samples, etc. further complicate the evaluation since it is assumed that the native concentrations are constant in triplicate analyses. In addition, concentrations of the target analytes in the sample can also far exceed spike amounts. If the native concentration of target analytes in the sample chosen for spiking is high relative to the spiking concentration, the differences in the native concentration between the unspiked sample and the spiked samples may also become very significant.

11.10.3 In addition to their reluctance to pay for MS and/or MSD analyses, most clients may not know what samples to collect for MS analysis. Clients may not collect sufficient samples, have no knowledge of the native analyte concentrations in the chosen sample, and may not have knowledge of MS spiking levels that are appropriate either for the sample or their project. For procedures with multiple target analytes, spiking levels may be inappropriate for one or more analytes, if samples that contain target analytes are chosen either in the field or by the laboratory. If no project samples are collected for MS analysis, samples from another project used for MS analysis may not be representative of the project samples. When project samples (e.g. surface soil) are chosen, their MS recovery characteristics may be different from those of other samples (e.g. sub-surface soil) in the same batch.

11.10.4 ACTION - If the sample chosen for MS/MSD is inappropriate, notify the client. When additional payments are authorized for re-analysis, re-extract and/or reanalyze the MS/MSD samples after employing cleanup

procedures (if applicable), dilution techniques to minimize matrix interference, or using other project samples that appear to be clean, when sufficient quantities are available. Re-spike the sample at a higher level (e.g., at approximately two to four times the sample concentration), then reanalyze the sample based on project-specific requirements. It is the responsibility of the client at all times to select appropriate samples for MS analysis, make payments for additional analyses, and to offer necessary guidance as needed to meet their data quality objectives.

11.10.5 ACTION - If the matrix effect cannot be resolved, discuss the situation in the case narrative. No action may be taken by the laboratory to qualify associated sample data based on MS/MSD recovery data alone. Professional judgment may be used by the user (client) in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

11.10.6 ACTION – No action is taken on MS/MSD recoveries data alone to qualify all associated samples. Since matrix interference could lead to lead to serious analytical problems, the supervisor should be consulted when significant outliers are noted. Document QC outliers for %recovery and RPD on the checklist and on client reports and draw attention to the QC outliers on the case narrative. No action may be taken on MS/MSD recovery data alone either to reanalyze samples and/or to qualify associated samples either by the analyst or by the end user. Professional judgment may be used by the user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

11.11 INTERNAL STANDARD PERFORMANCE – Internal Standard (IS) performance criteria ensures that the GC sensitivity and response is stable during each run.

11.11.1 IS area counts (use mean IS area from ICAL for the %D of IS area) must not vary by more than a factor of 2 (-50% or + 100%) from the associated ICAL/Calibration Verification standard. The internal standard areas for the CCV should not differ by a factor of 2 (-50% or + 100%) from the initial calibration of the same standard.

11.11.2 The retention time of the internal standard must not vary by more than 30 seconds from the associated daily calibration standard.

11.11.3 ACTION – If an internal standard is outside of QC limits of the associated daily standard, then re-analysis is required or a dilution may be required if it appears that interference's occur from target and non-target analytes at high concentrations relative to the ICAL. Do not dilute the sample to the point where all target analytes are non-detect. Professional judgement must be used when dilutions are required for IS criteria.

11.11.4 ACTION – If an internal standard is outside of QC limits of the associated ICAL or CCV standard, correct the problem and re-analyze samples with

a valid CCV. If re-analysis has been performed and it is still out, it is of the professional judgement of the analyst to dilute or not to dilute the sample further so as to not to dilute out any target analytes.

11.11.5 ACTION - Report results without qualification describing the QC problem in the case narrative. The user (client) may qualify positive results for compounds quantitated using the affected IS as estimated (J). If a very low IS area is reported, indicating a loss of sensitivity, non-detects may require qualification as invalid (I) or rejected (R). Document recommendation for the client in the case narrative.

11.11.6 ACTION – If an IS retention time varies by more than +/- 30 seconds, the total ion profile must be checked for any false positives or negatives. If a large shift has occurred, then professional judgement must be used as to whether reanalyze or not. See previous section for guidance.

11.12 TARGET COMPOUND IDENTIFICATION – Criteria for GC/ECD qualitative analysis is to minimize the number of erroneous positive or negative identifications of compounds.

11.12.1 Positive compounds must be within +/- 0.06 relative time (RRT) units of the standard RRT. The results should be >MDL or other threshold for reporting positives (e.g. LLR, RL) on both columns.

11.12.2 The RPD for sample amounts from the two columns for identified compounds should be within <40%. Document RPD calculations (See Appendix 6 for details) in Sample Result worksheets (Form-10 equivalents).

11.12.3 ACTION - If the RPD for positive results are <40%, report the result from the primary column without any flag.

11.12.4 ACTION - If the RPD is >40%, report the result from the primary column with a “J” flag. The result from the confirmation column may be reported with a “J” flag at the discretion of the analyst, if intererence is suspected for the compound of interest in the primary column.

11.12.5 ACTION – Professional judgement must be used when applying the criteria for GC/ECD analysis of target analytes. Take into account factors such as matrix effect and column bleed.

12.0 REFERENCES

12.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8081A, Organochlorine Pesticides by Gas Chromatography, December 1996.

12.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8081B, Organochlorine Pesticides by Gas Chromatography, Draft Revision 2A, May 1999.

12.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8082, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, December 1996.

12.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8082A, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Draft Revision 1A, May 1999.

12.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.

12.6 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

12.7 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3500B, Organic Extraction and Sample Preparation, December 1996.

12.8 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3520C, Continuous Liquid-Liquid Extraction, December 1996.

12.9 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3510C, Separatory Funnel Liquid-Liquid Extraction, December 1996.

12.10 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3511, Organic compounds in water by microextraction, Revision 0, November 2002.

12.11 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3540C, Soxhlet Extraction, December 1996.

12.12 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3541, Automated Soxhlet Extraction, December 1996.

12.13 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3545, Presuurized Fluid Extraction, December 1996.

12.14 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3550B, Ultrasonic Extraction, December 1996.

12.15 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3570, Microscale Solvent Extraction (MSE), Revision 0, November 2002.

12.16 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3600C, Cleanup, December 1996.

12.17 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3610B, Alumina Cleanup, December 1996.

12.18 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3611B, Alumina Column Cleanup and Separation of Petroleum Wastes, December 1996.

12.19 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3620B, Florisil Cleanup, December 1996.
12.20 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3620C, Florisil Cleanup, Revision 3, November 2000.
12.21 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3630C, Silica Gel Cleanup, December 1996.
12.22 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3640A, Gel-Permeation Cleanup, December 1996.
12.23 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3660B, Sulfur Cleanup, December 1996.
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12.26 Methods for the Determination of Organic Compounds in Drinking Water, Supplement II, EPA/600/4-88-039, Method 508, Determination of Chlorinated Pesticides in Water by Gas Chromatography with an Electron Capture Detector, July 1991.
12.27 Methods for the Determination of Organic Compounds in Drinking Water, Method 508.1, Determination of Chlorinated Pesticides in Water by Gas Chromatography with an Electron Capture Detector.
12.28 USEPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-media, Multi-concentration, OLM02.0.
12.29 USEPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-media, Multi-concentration, OLM03.0.
12.30 USEPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-media, Multi-concentration, OLM04.0.
12.31 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.
12.32 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.
12.33 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 3, January 2006.
12.34 AML Standard Operating Procedures, Q-Water-RC.
12.35 AML Standard Operating Procedures, Q-WasteMgmt-RD.
12.36 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
12.37 AML Standard Operating Procedures, Q-Controlcharts-RC.

Table 1

List of Default Target Compounds or Analytes for Organochlorine Pesticides Analysis (EPA Methods 508.1, 608, and 8081A)

Compound	CAS Registry No.
Aldrin	309-00-2
α-BHC	319-84-6
β-BHC	319-85-7
γ-BHC (Lindane)	58-89-9
δ-BHC	319-86-8
α-Chlordane	5103-71-9
γ-Chlordane	5103-74-2
Technical Chlordane	57-74-9
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan sulfate	1031-07-8
Endrin	72-20-8
Endrin aldehyde	7421-93-4
Endrin ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor epoxide	1024-57-3
Methoxychlor	72-43-5
Toxaphene	8001-35-2

Table 2

List of Optional Target Organochlorine Pesticide Analytes (EPA Methods 508.1, 608, and 8081A)

Compound	CAS Registry No.
Alachlor	15972-60-8
Captafol	2425-06-1
Chlorobenzilate	510-15-6
Chloroneb	2675-77-6
Chloropropylate	99516-95-7
Chlorothalonil	1897-45-6
DBCP*	96-12-8
DCPA	1861-32-1
Diallate*	2303-16-4
Dichlone	117-80-6
Dichloran	99-30-9
Dicofol	115-32-2
Etridiazole	2593-15-9
Halowax-1000	58718-66-4
Halowax-1001	58718-67-5
Halowax-1013	12616-35-2
Halowax-1014	12616-36-3
Halowax-1051	2234-13-1
Halowax-1099	39450-05-0
Hexachlorobenzene*	118-74-1
Hexachlorocyclopentadiene*	77-47-4
Isodrin*	465-73-6
Mirex	2385-85-5
Nitrofen	1836-75-5
PCNB	82-68-8
Permethrin	51877-74-8
Perthane	72-56-0
Propachlor	1918-16-7
Strobane	8001-50-1
trans-Nonachlor	39765-80-5
Trifluralin	1582-09-8

NOTE: Draft SW-846 Method 8081B contains the compounds marked with an asterisk superscript (*) in Table-1.

Table 3
Aqueous Sample MDL, LLR, MQL and DoD QSM Control and Marginal Exceedance (ME) Limits for LCS and MS Surrogate Recovery Limits

Chlorinated Pesticides Analysis													
Analytical Management Laboratories													
Preparation Methods:	3510C												
Analysis Methods:	8081A	8081B											
	MDL	**MDL**	**MDL**	**MDL**	**LLR**	**MQL**	**Ratio (RQ)**		**DoD QSM LCS & MS Control Limits**		**DoD QSM LCS & MS ME Limits**		**RPD for Dup.**
Compound	Spike	DB-5	DB-1701	Report	Report	Report	QL/DL	flag	Lower	Upper	Lower	Upper	Limit
	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L			**%R**	**%R**	**%R**	**%R**	%
α-BHC	0.025	0.0124	0.0087	**0.0124**	0.025	**0.10**	8.0		60	130	50	140	50
β-BHC	0.025	0.0221	0.0106	**0.0221**	0.025	**0.10**	4.5		65	125	55	135	50
γ-BHC	0.025	0.0154	0.0090	**0.0154**	0.025	**0.10**	6.5		25	135	10	155	50
δ-BHC	0.025	0.0036	0.0017	**0.0036**	0.025	**0.10**	27.9		45	135	30	150	50
Heptachlor	0.025	0.0176	0.0103	**0.0176**	0.025	**0.10**	5.7		40	130	30	145	50
Aldrin	0.025	0.0195	0.0096	**0.0195**	0.025	**0.10**	5.1		25	140	10	155	50
Heptachlor Epoxide	0.025	0.0210	0.0098	**0.0210**	0.025	**0.10**	4.8		60	130	50	140	50
γ-Chlordane	0.025	0.0201	0.0084	**0.0201**	0.025	**0.10**	5.0		60	125	50	135	50
Endosulfan I	0.025	0.0192	0.0090	**0.0192**	0.025	**0.10**	5.2		50	110	40	120	50
α-Chlordane	0.025	0.0202	0.0096	**0.0202**	0.025	**0.10**	5.0		65	125	55	135	50
Dieldrin	0.025	0.0163	0.0084	**0.0163**	0.025	**0.10**	6.2		60	130	50	140	50
DDE	0.025	0.0188	0.0072	**0.0188**	0.025	**0.10**	5.3		35	140	15	160	50
Endrin	0.025	0.0126	0.0186	**0.0186**	0.025	**0.10**	5.4		55	135	45	145	50
Endosulfan II	0.025	0.0154	0.0087	**0.0154**	0.025	**0.10**	6.5		30	130	10	150	50
Endrin Aldehyde	0.025	0.0123	0.0054	**0.0123**	0.025	**0.10**	8.1		55	135	40	150	50
DDD	0.025	0.0134	0.0103	**0.0134**	0.025	**0.10**	7.4		25	150	10	170	50
Endosulfan Sulfate	0.025	0.0117	0.0078	**0.0117**	0.025	**0.10**	8.5		55	135	40	150	50
DDT	0.025	0.0159	0.0095	**0.0159**	0.025	**0.10**	6.3		45	140	30	155	50
Endrin Ketone	0.025	0.0153	0.0082	**0.0153**	0.025	**0.10**	6.5		75	125	70	135	50
Methoxychlor	0.025	0.0258	0.0082	**0.0258**	0.025	**0.10**	3.9		55	150	40	165	50
Surrogates:													
DCB									30	135	30	135	
TCMX									25	140	25	140	
Criteria for the ratio (RD) of mean amount found to MDL: >1 but <5 and for MQL/MDL ratios (RQ) that are less than 3.													
QC limit for RPD is from the USACE document, Appendix I, EM 200-1-3, February 1, 2001.													
LCS (extended to the MS) %R limits are from the DoD QSM, Final Revision 2, June 2002.													
The following criteria will apply for marginal exceedances:													
Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).													
NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.													

Table 4
Soil Sample MDL, LLR, MQL and DoD QSM Control and Marginal Exceedance (ME) Limits for LCS and MS Surrogate Recovery Limits

Chlorinated Pesticides Analysis													
Analytical Management Laboratories													
Preparation Methods:	3550	3541											
Analysis Methods:	8081A	8081B											
	MDL	**MDL**	**MDL**	**MDL**	**LLR**	**MQL**	**Ratio (RQ)**		**DoD QSM LCS & MS Control Limits**		**DoD QSM LCS & MS ME Limits**		**RPD**
Compound	Spike	DB-5	DB-1701	Report	Report	Report	QL/DL	Flag	Lower	Upper	Lower	Upper	Limit
	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg			**%R**	**%R**	**%R**	**%R**	%
α-BHC	1.67	0.199	0.134	**0.199**	0.5	**1.67**	8.4		60	125	50	135	50
β-BHC	1.67	0.308	0.493	**0.493**	0.5	**1.67**	3.4		60	125	50	135	50
γ-BHC	1.67	0.227	0.253	**0.253**	0.5	**1.67**	6.6		60	125	50	135	50
δ-BHC	1.67	0.058	0.065	**0.065**	0.5	**1.67**	25.5		55	130	45	145	50
Heptachlor	1.67	0.228	0.327	**0.327**	0.5	**1.67**	5.1		50	140	35	155	50
Aldrin	1.67	0.306	0.424	**0.424**	0.5	**1.67**	3.9		45	140	30	155	50
Heptachlor Epoxide	1.67	0.328	0.481	**0.481**	0.5	**1.67**	3.5		65	130	55	140	50
γ-Chlordane	1.67	0.292	0.459	**0.459**	0.5	**1.67**	3.6		65	125	55	135	50
Endosulfan I	1.67	0.282	0.443	**0.443**	0.5	**1.67**	3.8		15	135	10	155	50
α-Chlordane	1.67	0.292	0.475	**0.475**	0.5	**1.67**	3.5		65	120	55	130	50
Dieldrin	1.67	0.241	0.408	**0.408**	0.5	**1.67**	4.1		65	125	55	135	50
DDE	1.67	0.281	0.406	**0.406**	0.5	**1.67**	4.1		70	125	60	135	50
Endrin	1.67	0.266	0.348	**0.348**	0.5	**1.67**	4.8		60	135	50	145	50
Endosulfan II	1.67	0.269	0.406	**0.406**	0.5	**1.67**	4.1		35	140	20	160	50
Endrin Aldehyde	1.67	0.299	0.464	**0.464**	0.5	**1.67**	3.6		35	145	20	165	50
DDD	1.67	0.338	0.389	**0.389**	0.5	**1.67**	4.3		30	135	10	155	50
Endosulfan Sulfate	1.67	0.175	0.293	**0.293**	0.5	**1.67**	5.7		60	135	50	145	50
DDT	1.67	0.260	0.268	**0.268**	0.5	**1.67**	6.2		45	140	30	155	50
Endrin Ketone	1.67	0.259	0.440	**0.440**	0.5	**1.67**	3.8		65	135	55	145	50
Methoxychlor	1.67	0.314	0.439	**0.439**	0.5	**1.67**	3.8		55	145	45	155	50
Surrogates:													
DCB									55	130	55	130	
TCMX									70	125	70	125	
Criteria for the ratio (RD) of mean amount found to MDL: >1 but <10 (for soil) and for MQL/MDL ratios (RQ) that are less than 3.													
QC limit for RPD is from the USACE document, Appendix I, EM 200-1-3, February 1, 2001.													
LCS (extended to the MS) %R limits are from the DoD QSM, Final Revision 2, June 2002.													
The following criteria will apply for marginal exceedances:													
Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).													
NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.													

Table 5
List of Appendices

Appendix Number	Appendix Topic
1	Target Compound List and their Concentrations In the Stock Standard
2	Retention Time Summary DDT and Endrin Breakdown Summary
3	Initial Calibration (ICAL) Summary (Form-6 equivalents) Instrument Printouts
4	Calibration Verification (ICV and CCV) Summary (Form-7 equivalents) Instrument Printouts and Standard Chromatograms
5	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
6	Sample Result Worksheets (Form-10 equivalents)
7	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
8	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
9	Surrogate Recovery Reports (Form-2 equivalents)
10	Method Detection Limit (MDL) Study Summary
11	QC Association Forms (Form-4 equivalents) Sample Preparation Logsheets Instrument Run Log
12	Sample Reports (Form-1 equivalents)
13	USACE Method Quality Objectives for Method 8081A AFCEE MQOs for Method 8081A DoD QSM MQOs and Limits for percent recoveries

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. O-PCBs-RC

POLYCHLORINATED BIPHENYLS BY GC/ECD (EPA 508.1, 608 and SW846-8082A)

October 12, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure, which is based on EPA methods (508.1, 608, and SW-846 Method 8082 and 8082A) is used to determine the concentrations of polychlorinated biphenyls (PCBs) as Aroclors in extracts from solid and aqueous matrices. Open-tubular, capillary columns are employed with electron capture detectors (ECD). When compared to packed columns, these fused-silica, open-tubular columns offer improved resolution, better selectivity, increased sensitivity, and faster analysis. The target compounds listed below may be determined by either a single- or dual-column analysis system.

Compound	CAS Registry No.
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5

1.2 Aroclors are multi-component mixtures. When samples contain more than one Aroclor, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of Aroclors that have been subjected to environmental degradation ("weathering") or degradation by treatment technologies. Such weathered multi-component mixtures may have significant differences in peak patterns than those of Aroclor standards.

1.3 Quantitation of PCBs as Aroclors is appropriate for many regulatory compliance determinations, but is particularly difficult when the Aroclors have been weathered by long exposure in the environment.

1.4 Another approach based on PCB congeners may afford greater quantitative accuracy when PCBs are known to be present and it is of particular value in determining weathered Aroclors. However, the congener method may not be useful when regulatory requirements are based on Aroclor concentrations. The laboratory does not employ the congener approach for the analysis of PCBs.

1.5 Compound identification based on single-column analysis should be confirmed on a second column, or should be supported by at least one other qualitative technique. This SOP describes analytical conditions for a second gas chromatographic column that can be used to

confirm the measurements made with the primary column. GC/MS (SW-846 Method 8270) can also be used as a confirmation technique when sensitivity permits.

1.6 This SOP describes the dual-column option, allowed by the method. The option allows a hardware configuration of two analytical columns joined to a single injection port. The option allows one injection to be used for dual-column analysis. Analysts are cautioned that the dual-column option may not be appropriate when the instrument is subject to mechanical stress, many samples are to be run in a short period, or when highly contaminated samples are analyzed.

1.7 The analyst must select columns, detectors and calibration procedures most appropriate for the specific analytes of interest in a study. Matrix-specific performance data must be established and the stability of the analytical system and instrument calibration must be established for each analytical matrix (e.g., hexane solutions from sample extractions, diluted oil samples, etc.). Example chromatograms and GC conditions are provided as guidance.

1.8 The MDLs for Aroclors vary in the range of 0.054 to 0.90 µg/L in water and 57 to 70 µg/kg in soils. MDLs and MQLs for the laboratory are shown in Tables 1 and 2.

1.9 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs (GC) and skilled in the interpretation of gas chromatograms.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample (approximately 1 L for liquids, 2-g to 30-g for solids) is extracted using the appropriate matrix-specific sample extraction technique.

2.2 Aqueous samples are extracted at neutral pH with methylene chloride using Method 3510 (separatory funnel), Method 3520 (continuous liquid-liquid extractor), Method 3535 (solid-phase extraction) or other appropriate technique.

2.3 Solid samples are extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) using AML SOPs based on EPA SW-846 Method 3540 (Soxhlet), Method 3541 (automated Soxhlet), Method 3550 (sonication) or other appropriate technique. The SW-846 methods should be used when a suitable AML SOP is unavailable during method development and/or implementation.

2.4 Tissue samples may be extracted using Method 3562 (supercritical fluid extraction) or other techniques such as those described in Section 2.3 for solid samples.

2.5 Extracts for PCB analysis may be subjected to a sulfuric acid cleanup or a sulfuric acid/potassium permanganate cleanup (AML SOP based on EPA SW-846 Method 3665) designed specifically for these analytes. The SW-846 methods should be used when a suitable AML SOP is unavailable during method development and/or implementation. A summary of the sulfuric acid cleanup procedure used by AML is included in this SOP. This cleanup technique will remove (destroy) many single component organochlorine or organophosphorus pesticides. Therefore, these extracts

may not be used for the analysis of organochlorine pesticide compounds, if these cleanups are performed.

2.5 After cleanup, the extract is analyzed by injecting a 2-uL aliquot into a gas chromatograph with a narrow- or wide-bore fused silica capillary column and electron capture detector (GC/ECD).

2.6 The chromatographic data is used to determine the seven Aroclors listed in Section 1.1.

3.0 INTERFERENCES

3.1 Refer to AML SOPs based on SW-846 Methods 3500, 3600, and 8000 for a discussion of interferences. The SW-846 methods should be used when a suitable AML SOP is unavailable during method development and/or implementation. A summary of these procedures is included in this SOP.

3.2 Interferences co-extracted from the samples will vary considerably from matrix to matrix. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation. Sources of interference in this method can be grouped into three broad categories.

3.2.1 Contaminated solvents, reagents, or sample processing hardware.

3.2.2 Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.

3.2.3 Compounds extracted from the sample matrix to which the detector will respond (e.g. chlorinated pesticdes and herbicides).

3.3 Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determinations.

3.3.1 Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination.

3.3.2 Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

3.3.3 These materials can be removed through the use of AML SOP based on EPA SW-846 Method 3665 (sulfuric acid/permanganate cleanup). The SW-846 methods should

be used when a suitable AML SOP is unavailable during method development and/or implementation. A summary of these procedures is included in this SOP.

3.4 Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water. Drain the glassware, and dry it in an oven at 130°C for several hours, or rinse with methanol and drain. Store dry glassware in a clean environment.

NOTE: Oven-drying of glassware used for PCB analysis can increase contamination because PCBs are readily volatilized in the oven and spread to other glassware. Therefore, exercise caution, and do not dry glassware from samples containing high concentrations of PCBs with glassware that may be used for trace analyses.

3.5 Elemental sulfur (S_8) is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. Sulfur can be removed through the use of AML SOP based on EPA SW-846 Method 3660. The SW-846 methods should be used when a suitable AML SOP is unavailable during method development and/or implementation.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph - An analytical system complete with gas chromatograph suitable for on-column and split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and recorder/integrator or data system: Two Hewlett Packard (Agilent) Model 5890 Series II equipped with dual ECD detectors, and two Agilent autosamplers (Model 7673/7673A).

4.2 GC columns - A dual-column approach is used, which involves a single injection that is split between two columns that are mounted in a single injector in a single gas chromatograph. This dual-column approach employed at AML consists of two 0.25 mm ID columns.

4.2.1 Narrow-bore columns that are installed in split/splitless (Grob-type) injectors. Column pair is mounted in a press-fit Y-shaped glass 3-way union splitter (J&W Scientific, Catalog No. 705-0733) or a Y-shaped fused-silica connector (Restek, Catalog No. 20405), or equivalent.

4.2.1.1 30 m x 0.25 mm ID fused silica capillary column chemically bonded with SE-54 (DB-5 or equivalent), 1 µm film thickness.

4.2.1.2 30 m x 0.25 mm ID fused silica capillary column chemically bonded with 14% cyanopropylmethylpolysiloxane (DB-1701, or equivalent), 1.0 µm film thickness.

4.3 Column rinsing kit - Bonded-phase column rinse kit (J&W Scientific, Catalog No. 430-3000), or equivalent.

4.4 Volumetric flasks - 10-mL and 25-mL, for preparation of standards.

5.0 REAGENTS

5.1 Reagent grade or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate standards) at 4 degrees C in polytetrafluoroethylene (PTFE)-sealed containers in the dark. When a lot of standards is prepared, it is recommended that aliquots of that lot be stored in individual small vials. All stock standard solutions must be replaced after one year or sooner if routine QC indicates a problem. All other standard solutions must be replaced after six months or sooner if routine QC indicates a problem.

5.2 Sample extracts prepared by AML SOPs based on SW-846 Methods 3510, 3520, 3540, 3541, 3545, or 3550 need to undergo a solvent exchange step prior to analysis. The following solvents are necessary for dilution of sample extracts. All solvent lots should be pesticide quality or equivalent and should be determined to be phthalate-free.

5.2.1 n-Hexane, C_6H_{14}

5.2.2 Isooctane, $(CH_3)_3CCH_2CH(CH_3)_2$

5.3 The following solvents may be necessary for the preparation of standards. All solvent lots must be pesticide quality or equivalent and should be determined to be phthalate-free.

5.3.1 Acetone, $(CH_3)_2CO$

5.3.2 Toluene, $C_6H_5CH_3$

5.4 Organic-free reagent water.

5.5 Stock standard solutions - The stock standard solutions are purchased from Ultra Scientific as certified solutions. Second source standards are purchased from Supelco.

5.5.1 The surrogate 2,4,5,6-Tetrachloro-m-xylene (TCMX) is purchased from Ultra (cat # IST-440) at a concentration of 2,000-ug/mL.

5.5.2 The surrogate, decachlorobiphenyl is purchased from Ultra (cat # PPS-150) at a concentration of 1,000-ug/mL.

5.5.3 The surrogate spiking solution containing TCMX and DCB at a concentration of 200-ug/mL each is purchased from Ultra (Cat # ISM-320).

5.5.4 The Aroclor-1016/1260 mixture is purchased from Ultra (cat # PPM-8082) at a concentration of 1,000-ug/mL each in hexane:toluene (1:1).

5.5.5 Individual standards of the various Aroclors are also purchased at a concentration of 1,000-ug/ml in iso-octane:

5.5.5.1 Aroclor-1016, catalogue #EPA-1282
5.5.5.2 Aroclor-1221, catalogue # EPA-1292
5.5.5.3 Aroclor-1232, catalogue # EPA-1302
5.5.5.4 Aroclor-1242, catalogue # EPA-1312
5.5.5.5 Aroclor-1248, catalogue # EPA-1342
5.5.5.6 Aroclor-1254, catalogue # EPA-1352
5.5.5.7 Aroclor-1260, catalogue # EPA-1362
5.5.5.8 Aroclor-1262, catalogue # EPA-1372
5.5.5.9 Aroclor-1268, catalogue # EPA-1382

5.5.6 Second source Aroclor-1016/1260 (at 500-ug/mL each) mixture containing the two surrogates (at 50-ug/mL each) is purchasd from Supelco (46846-u) for the preparation of initial calibration verification (ICV) standards, which are used for compliance with USACE requirements.

5.5.7 Second source individual Aroclor solutions (1000-ug/mL in isooctane) are purchased from Supelco:

5.5.7.1 Aroclor-1016, catalogue #48097
5.5.7.2 Aroclor-1221, catalogue #48098
5.5.7.3 Aroclor-1232, catalogue #44805
5.5.7.4 Aroclor-1242, catalogue #44806
5.5.7.5 Aroclor-1248, catalogue #44807
5.5.7.6 Aroclor-1254, catalogue #44808
5.5.7.7 Aroclor-1260, catalogue #44809
5.5.7.8 Aroclor-1262, catalogue #44810

5.5.8 Internal Standard (IS) - The compound, 1-bromo-2-nitrobenzene is used as an internal standard, which is added to each sample extract prior to analysis, and included in each of the initial calibration standards. The stock standard solution is purchased through Ultra (cat # PPS-351) at a concentration 5000-ug/ml.

5.5.8.1 An intermediate IS solution is prepared by diluting 1.0-mL of the stock solution (5000-ug/mL) with 99-mL of iso-octane for a final concentration of 50-ug/mL or 50,000-ng/mL.

5.5.8.2 An aliquot (10-uL) of this solution is spiked 1.0-ml of sample extracts and calibration standards for a final IS concentration of 500-ng/mL in the solution injected into the gas chromatograph.

5.6 Calibration standards for Aroclors

5.6.1 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 and 1260 at five concentrations should be sufficient to demonstrate the linearity of the detector response without the necessity of performing initial calibrations for each of the seven Aroclors. In addition, such a mixture can be used as a standard to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample.

5.6.2 Default AR1016/1260 Multi-point Initial Calibration (ICAL) Standards - Using standards obtained from Ultra, prepare a minimum of five calibration standards containing the two surrogates (five different levels), the internal standard (one level) and equal concentrations of both Aroclor 1016 and Aroclor 1260 (five different levels).

5.6.2.1 Dilute 200-uL of the 1,000-ug/mL mixed Aroclor 1016/1260 standard (Ultra PPM-8082), 100-uL of the mixed surrogate standard (ISM-320) to 100-ml with acetone for a final concentration of 2000 ng/ml of each Aroclor and 200-ng/mL for each surrogate. This solution expires one year from the date of preparation. Replace sooner if signs of degradation is observed.

5.6.2.2 The 2000 ng/ml concentration standard (Section 5.6.2.1) is the high point of the calibration for the Aroclors. Add 10-uL of the IS intermediate solution (Section 5.5.8.1) to 1-mL of the 2000-ng/mL standard in a 1-mL GC injection vial.

5.6.2.3 From the 2000-ng/ml standard, 400-uls is diluted to 1.0 ml for a final concentration of 800-ng/ml. Add 10-uL of the IS intermediate solution (Section 5.5.8.1) to 1-mL of the 800-ng/mL standard in a 1-mL GC injection vial.

5.6.2.4 From the 2000-ng/ml standard, 300-uls is diluted to 1.0 mls for a final concentration of 600-ng/ml. Add 10-uL of the IS intermediate solution (Section 5.5.8.1) to 1-mL of the 600-ng/mL standard in a 1-mL GC injection vial.

5.6.2.5 From the 2000-ng/ml standard, 200-uls is diluted to 1.0 mls for a final concentration of 400-ng/ml. Add 10-uL of the IS intermediate solution (Section 5.5.8.1) to 1-mL of the 400-ng/mL standard in a 1-mL GC injection vial.

5.6.2.6 From the 2000-ng/ml standard, 100-uls is diluted to 1.0 mls for a final concentration of 200-ng/ml. Add 10-uL of the IS intermediate solution (Section 5.5.8.1) to 1-mL of the 200-ng/mL standard in a 1-mL GC injection vial.

5.6.2.7 The respective concentration of the surrogates in the five calibration standard solutions are as follows: 200-ng/mL, 80-ng/mL, 60-ng/mL, 40-

ng/mL, and 20-ng/mL. The internal standard concentration is 500-ng/mL in each of the ICAL standard solutions.

5.6.2.8 Injection vials containing the five point calibration standards are prepared daily.

5.6.3 Single standards of each of the other five Aroclors are required to aid the analyst in pattern recognition. Assuming that the Aroclor 1016/1260 standards described in Sec. 5.6.2 have been used to demonstrate the linearity of the detector, these single standards of the remaining five Aroclors are also used to determine the calibration factor for each Aroclor. Prepare a daily calibration standard for each of these Aroclors at a concentration, which should correspond to the mid-point of the linear range of the detector (1000 ng/ml).

5.6.3.1 Dilute 100-uL of the 1,000-ug/mL Aroclor standard from Ultra (See Section 5.5.5.2 to 5.5.5.6) and 100-uL of the mixed surrogate standard (ISM-320) to 100-ml with acetone for a final concentration of 1000 ng/ml of each Aroclor (present in individual solutions) and 200-ng/mL for each surrogate. The intermediate solution should be replaced six months from the date of preparation. Replace sooner if signs of degradation is observed.

5.6.3.2 Add 10-uL of the IS intermediate solution (Section 5.5.8.1) to 1-mL of the 1000-ng/mL standard in a 1-mL GC injection vial.

5.6.3.3 The working standards (Section 5.6.3.2 and 5.6.3.4) are prepared daily, whenever a new ICAL curve is generated.

5.6.3.4 Prepare individual single concentration standards of Aroclor-1262 (Section 5.5.5.8) and Aroclor-1268 (Section 5.5.5.9) for special projects as described in Section 5.6.3.1. These Aroclors are not standard target analytes.

5.6.4 Custom Aroclor Multi-point Initial Calibration (ICAL) Standards - For projects that require multi-point calibration with the Aroclor of concern, prepare a minimum of five calibration standard solution using the specified Aroclor instead of the AR1016/1260 mixture. The Aroclor 1016/1260 standards need not be used if a specific Aroclor is known to be present in site samples. Refer to the project specific Quality Assurance Project Plan for specific information.

5.6.4.1 The intermediate and working calibrations standards are prepared similiarly to 5.6.2.1 to 5.6.2.8. Aroclor stock standards described in Sections 5.5.5.1 to 5.5.5.9 are used.

5.6.5 Initial Calibration verification (ICV) - The ICV solution is prepared from a second source of stock standard solutions.

5.6.5.1 ICV for default ICAL - Prepare an ICV intermediate solution from the Supelco standard (See Section 5.5.6), which contains the two default ICAL target analytes (Aroclor-1016 and -1260) at a concentration of 500-ug/mL and the two surrogates at 50-ug/mL. Dilute 200-uL of the Supelco standard to 100-ml with acetone for a final concentration of 1000 ng/ml of each Aroclor and 100-ng/mL for each surrogate. This

solution expires one year from the date of preparation. Replace sooner if signs of degradation is observed.

5.6.5.2 Add 10-uL of the IS intermediate solution (Section 5.5.8.1) to 1-mL of the 1000-ng/mL ICV intermediate standard in a 1-mL GC injection vial.

5.6.5.3 The working standard (Section 5.6.5.2) is prepared daily, whenever a new ICAL curve is generated.

5.7 Internal standard

5.7.1 The SW-846 recommeds the use of Decachlorobiphenyl (DCB) as the internal standard for PCB congener analysis. Although the use of an internal standard is not required for the analysis of Aroclors, AML uses the compound, 1-bromo-2-nitrobenzene as an internal standard (IS), to keep parity with other EPA determinative methods (e.g. 8081A and 8081B).

5.7.2 The internal standard is added to each sample extract prior to analysis, and included in each of the initial calibration standards. An aliquot (10-uL) of the IS solution is used to spike 1.0-mL of the sample extracts and calibration standards for a final concentration of 500-ng/ml in the solution injected into the GC.

5.8 Surrogate Sample Spiking Solutions

5.8.1 The SW-846 recommeds the use of Decachlorobiphenyl as the surrogate for PCBs analysis. The AML procedure involves sharing of a sample extract for the analysis of organochlorine pesticides (8081) and PCBs (8082), which are method analytes for other EPA methods (508.1 and 608). Both DCB and tetrachloro-m-xylene (TCMX) are used as surrogates, and they are added to each sample prior to extraction. The stock surrogate solution is purchased through Ultra Scientific (Catalog # ISM-321X) at a concentration of 200 ng/mL. Spike 1.0-ml of this solution to each sample including QC samples prior to extraction. The final extract (5-ml final volume) concentration will be 40-ng/mL.

5.9 LCS and MS Spike Standards

5.9.1 The LCS spiking solution is prepared from the source of stock standard solution (Ultra cat # PPM-8082) that is used for ICAL. Dilute 300-uL of this solution to 100-mL with acetone. The resulting matrix spike solution is at a concentration of 3,000-ng/mL. Spike each LCS and LCSD sample with 1.0-mL of this 3000-ng/mL standard. The final extract (5-ml final volume) concentration will be 600-ng/mL in each of the two Aroclors (1016 and 1260).

5.9.2 The MS spiking solution is identical to the LCS spiking solution. Spike each MS and MSD sample with 1.0-mL of this 3000-ng/mL standard. The final extract (5-ml final volume) concentration will be 600-ng/mL in each of the two Aroclors (1016 and 1260).

6.0 SAMPLE PRESERVATION HANDLING & STORAGE

6.1 One litre amber glass bottles with Teflon lined screw caps may be used for collecting aqueous samples in accordance with EPA SW-846 method 8082. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 2 bottles for each water sample to be collected. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. See project specific field sampling plan (FSP) for sample collection and preservation instructions. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.

6.2 One 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples in accordance with EPA SW-846 Method 8082. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each solid sample. Quality control samples such as MS and MSD should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. These samples may be shared with samples collected for other analyses such as semivolatiles (8270C), chlorinated pesticides (8081A), diesel (DRO, 8015-mod.) and other pesticides (8140, etc.).

6.3 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction. Analytical data generated outside of the specified holding times must be considered to be minimum values only. The holding time for the extraction of aqueous samples is 7 days from the date of sample collection. The holding times for the extraction of soil, sediment and waste samples is 14 days from the date of sample collection. The holding time for the instrumental analysis of sample extracts is 40 days from the date of extraction.

7.0 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control contain a number of regulated compounds at high concentrations. The toxicity of these compounds present in such complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Aqueous and soil samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters after the solvent(s) have been allowed to evaporate in a hood.

9.0 **PROCEDURE**

9.1 Sample extraction – Aqueous Samples

9.1.1 Refer to specific AML extraction SOPs based on EPA SW-846 Method 3500B for guidance in choosing the appropriate extraction procedure. The SW-846 methods including the overview (Method 3500B) should be used when a suitable AML SOP is unavailable during method development and/or implementation. The microextraction procedures described in the SOP on organochlorine pesticides analysis may also be used for rapid TAT projects in support of remediation efforts. A summary of the SW-846 aqueous extraction methods and associated AML SOPs are included in this SOP.

9.1.2 Manual Liquid/Liquid Extraction (Method 3510C) - In general, water samples are extracted at a neutral pH with methylene chloride using a separatory funnel (AML SOP based on SW-846 Method 3510C) or a continuous liquid-liquid extractor (Method 3520C, not used at AML).

9.1.2.1 Using a black marker, mark the meniscus of the water in the sample container. Pour the entire contents of the container into a 2-liter separatory funnel. Rinse the sample container with 3 aliquots of 20 mls methylene chloride and place the rinse in the separatory funnel. Add distilled water to the sample container up to the marked meniscus. Pour the distilled water into a 1-liter graduated cylinder. Record this measured volume in the Sample Extraction Log Book. Using pH indicating paper (0-14) record the pH of the sample (sample should be at a neatral pH). This is accomplished by dipping the tip of a Pasteur pipette into the water and placing the droplets of water on the pH paper. Never place the pH paper into the water sample. Add 1 mL of surrogate solution to all of the samples (including QC) and 1.0-ml of appropriate spiking solution to the LCS/LCSD & MS/MSD. Adjust, if necessary, the pH to 7 using either 50/50 sulfuric acid solution or 10N sodium hydroxide solution.

9.1.2.2 An alternate procedure, which may be employed to measure out water samples is described below. Using a 1-Liter graduated cylinder, measure out 1.00 liter (or lower volumes as low as 100-mL for high concentration samples) of the sample, and transfer it to a 2000-mL separatory funnel. Record the exact volume in the

organic Extraction logbooks. This alternate procedure may be used to measure out lower sample volumes for the re-extraction of heavily contaminated samples with surrogate recoveries outside the QC limits, which are diluted out during analysis. This procedure may also be used to measure out smaller aliquots (e.g. 500-mL) of the sample for MS/MSD analysis, when additional sample containers were not collected for these analyses.

9.1.2.3 Add 60-mLs of methylene chloride to the separatory funnel and shake for 2 minutes. Take care to vent the separatory funnel to relieve excess pressure. Allow the organic layer to separate from the water layer for a minimum of 10 minutes. Drain the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus. Repeat the extraction 3 more times using 60 mLs of methylene chloride.

9.1.2.4 Add two boiling chips to the K-D flask and attach a three ball Snyder column. Prewet the Snyder column with 1 mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1-ml to 5-ml, add 5-ml of iso-octane or hexane and allow sample to come back down. Remove the K-D flask and allow cooling for at least 10 minutes.

9.1.2.5 Concentrate the extract by nitrogen blow-down to 1.0-mL making sure **all** methylene chloride has been removed. Adjust sample to 5.0-ml and proceed to analyze by GC/ECD. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 500-ng/mL. NOTE: When sample extracts prepared for organochlorine pesticides are used to screen for PCBs, the internal standard concentration that is used is 50-ng/mL in the final extract and in calibration solutions.

9.2 Sample extraction – Soil Samples: Solid samples are extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) using one of the Soxhlet extraction (AML SOPs based on Method 3540C or 3541) procedures, ultrasonic extraction (AML SOP based on Method 3550B), or other appropriate procedure. Use of hexane-acetone for extraction, which is also used for the extraction of soil samples for organochlorine pesticides generally reduces the amount of interferences that are extracted and improves signal-to-noise. The microextraction procedures described in the SOP on organochlorine pesticides analysis may also be used for rapid TAT projects in support of remediation efforts. A summary of the SW-846 soil extraction methods and associated AML SOPs are included in this SOP.

9.2.1 Sample Extraction by Sonication – SW-846 Method 3550

9.2.1.1 Add 30-g of soil to a 500 mL beaker. Record the exact weight in the organic Extraction Log Book. Add enough sodium sulfate to dry the soil and mix well with a spoon. Add 1.0 mL of surrogate solution to all the samples (including QC) and 1.0-ml of appropriate spike spiking solutions to the LCS/LCSD & MS/MSD samples.

9.2.1.2 Add 50- to 100-mLs of the aceone:methylene chloride (20:80) mixture to the beaker and mix the contents with a spoon for 1 minute. Sonicate the soil/methylene chloride mixture for 3 minutes ensuring that the soil is free flowing and not in clumps. Decant the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus.

9.2.1.3 Extract the sample twice more with additional 50- to 100-mL aliquots of the solvent mixture. The solution should be sonicated for 3 minutes each time. Decant the solution in to the same K-D as described above.

9.2.1.4 Add two boiling chips to the K-D flask and attach a three ball Snyder column. Prewet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1-mL to 5-mL, add 5-mL of iso-octane or hexane and allow sample to come back down. Remove the K-D flask and allow to cool for at least 10 minutes.

9.2.1.5 Concentrate the extract by nitrogen blow-down to 1.0 mLs making sure **all** methylene chloride as been removed. Adjust sample to 5-ml and proceed to analyze by GC/ECD if no cleanup procedures are required. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 500-ng/µL. NOTE: When sample extracts prepared for organochlorine pesticides are used to screen for PCBs, the inetrnal standard concentration that is used is 50-ng/mL in the final extract and in calibration solutions.

9.2.2 Sample Extraction by Automated Soxhlet Procedure A – SW-846 Method 3541

9.2.2.1 Automated Soxhlet Extraction Procedure-A involves IKA equipment, which are capable of handling 30-g sample aliquots. The set-up consists of six 4-position extractors to handle a total of 24 samples, which will include up to 5 QC samples (MB, LCS, LCSD, MS, and MSD). The six extractors, equipped with independent heating and cooling systems are controlled by a single personal computer. Follow manufacturer instructions for setting up and operating the IKA Soxhlet equipment.

9.2.2.2 Weight out 30-g of the soil sample to three significant numbers (±0.1-g) into 500-mL beakers. Record the exact weight in the organic Extraction logbook (See Appendix 11 for documentation). Add enough sodium sulfate (60-g) to dry the soil and mix well with a spoon. When the soil is dry (freely flowing powder), the sample is ready for Soxhlet extraction.

9.2.2.3 Clean and completely assemble the IKA equipment. Raise the condenser from the top vessel. Assemble the top vessel with the stainless steel filter base and a new disposable Teflon micron-sized filter paper. Transfer the soil/drier mixture quantitatively to the top vessel. Add 1.0-mL of surrogate solution to all the samples (including QC) and 0.25-mL of the appropriate spiking solutions to the LCS/LCSD & MS/MSD samples. Add 100-mL of the solvent mixture (methylene chloride:acetone 80:20) to the bottom vessel and tightly screw the top vessel on to the bottom vessel containing solvent. Replace the condenser unit on the top vessel. Turn the unit on and allow the unit to cycle (45-minutes). Completely drain the solvent from the sample chamber using the instrument cycle control mechanism. Repeat the extraction process twice using the same solvent contained in the bottom vessel. Decant the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus.

9.2.2.4 Add two boiling chips to the K-D flask and attach a three-ball Snyder column. Prewet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1-ml, add 5-mL of hexane and continue with solvent removal. When the extract reaches 1 to 2-mL remove the K-D flask and allow to cool for at least 10 minutes.

9.2.2.5 Quantiatively transfer the extract to a 10-mL culture tube equipped with a PTFE-lined cap with graduation at 5-mL. Adjust the final volume to 5-mL with hexane. Proceed to analyze by GC/ECD if no cleanup procedures are required. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 50-ng/µL.

9.2.3 Sample Extraction by Automated Soxhlet Procedure B – SW-846 Method 3541

9.2.3.1 Automated Soxhlet Extraction Procedure-B involves Tecator Soxtec equipment, which are capable of handling only 10-g sample aliquots. The set-up consists of three 6-position extractors and two 2-position extractors to handle a total of 22 samples, which will

include up to 5 QC samples (MB, LCS, LCSD, MS, and MSD). The five extractors are operated independent using three hot oil baths equipped with independent controllers. The condensers are cooled using a circulating refrigerant bath. Follow manufacturer instructions for setting up and operating the Soxtec equipment.

9.2.3.2 Weight out 10-g of the soil sample to three significant numbers (±0.1-g) into 500-mL beakers. Record the exact weight in the organic Extraction logbook (See Appendix 11 for documentation). Add enough sodium sulfate (10-g) to dry the soil and mix well with a spoon. When the soil is dry (freely flowing powder), transfer the entire contents of the beaker quantitatively to the rigid paper thimbles suspended from the stainless steel holders. Add 1.0-mL of surrogate solution to all the samples (including QC) and 0.25-mL of the appropriate spiking solutions to the LCS/LCSD & MS/MSD samples.

9.2.3.3 Clean and completely assemble the Soxtec extractors. Check the heating oil level in the automated Soxhlet unit and add oil if needed. See service manual for details. Set the temperature on the service unit at 80°C. Press the “MAINS” button and verify that the switch lamp is now in the “ON” position. Set the flow rate for the circulator at approximately 2-L/min to minimize solvent loss from the condensing unit.

9.2.3.4 Immediately transfer the thimbles containing the weighed samples into the condensers. Raise the knob to the "BOILING" position. The magnet will now fasten to the thimble. Lower the knob to the "RINSING" position. The thimble will now hang just below the condenser valve.

9.2.3.5 Insert the extraction cups containing boiling chips, and load each with 50-mL of extraction solvent. Using the cup holder, lower the locking handle, ensuring that the safety catch engages. The cups are now clamped into position. (The seals must be pre-rinsed or pre-extracted with extraction solvent prior to initial use).

9.2.3.6 Move the extraction knobs to the "BOILING" position. The thimbles are now immersed in solvent. Set the timer for 60 minutes. The condenser valves must be in the "OPEN" position. Extract for the preset time.

9.2.3.7 After rinse time has elapsed, close the condenser valves by turning each a quarter-turn, clockwise. When all but 2- to 5-mL of solvent have been collected, open the system and remove the cups.

9.2.3.8 Transfer the contents of the cups to 15-mL graduated, conical-bottom glass tubes attached to the K-D flasks. Rinse the cups using methylene chloride and add the rinsates to the glass tubes.

9.2.3.9 Add two boiling chips to the K-D flask and attach a three-ball Snyder column. Pre-wet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so

that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1-ml, add 5-mL of hexane and continue with solvent removal. When the extract reaches 1 to 2-mL remove the K-D flask and allow to cool for at least 10 minutes.

9.2.3.10 Quantiatively transfer the extract to a 10-mL culture tube equipped with a PTFE-lined cap with graduation at 5-mL. Adjust the final volume to 5-mL with hexane. Proceed to analyze by GC/ECD if no cleanup procedures are required. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 50-ng/µL.

9.2.4 Sample Preparation for waste samples

9.2.4.1 If the waste is soluble in extraction solution, add approximately 1.0-g to a 15-mL culture tube. Record the exact weight in the Organic Extraction Log. Add 1.0-mL of the surrogate solution for samples and 1.0mL of surrogate plus 1.0-mL of appropriate spiking solution for LCS/LCSD & MS/MSD samples. Dilute the extract to 10.0 mLs with extraction solution and proceed with GC/ECD analysis. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 500-ng/µL.

9.2.4.2 If the waste is not soluble in the extraction solution, the sample must be extracted like a sediment/soil sample using only 1.0-g instead of 30-g. Proceed to cleanup steps if the sample appears to be dirty.

9.2.5 Spiking Solutions - When the presence of specific Aroclors is not anticipated, the Aroclor 1016/1260 mixture is used for spiking LCS, LCSD, MS and MSD samples. When specific Aroclors have been identified in site samples, calibration solutions and spiking solutions based on the specifc Aroclor may be used for the analysis. Procedues described above should be used with appropriate modification in such instances.

9.3 Extract cleanup - The cleanup options that are available for PCBs analysis have been described earlier. A summary of the sulfuric acid cleanup procedure (See AML SOP based on SW-846 Method 3665A) is described in this SOP.

9.3.1 Transfer 2.0-mL of the sample extract using a gas-tight syringe to a 10-mL culture tube equipped with a Teflon-lined screw cap. Add 5.0-mL of concentrated sulfuric acid, cap it, shake, and centrifuge.

9.3.2 If color persits, transfer 1.5-mL of the sample extract after acid cleanup to another 10-mL culture tube and treat it with 5-mL of potassium permanganate solution.

9.3.3 Transfer 1.0-mL of the upper hexane layer using a gas-tight syringe to a clean test tube. Fortify the extract with internal standard and analyze as described elsewhere.

9.4 GC conditions - This SOP based on the SW-846 Method 8082 and 8082A, which allows the analyst to choose between a single-column or a dual-column configuration in the injector port. Either wide- or narrow-bore columns may be used.

9.4.1 Single-column analysis - This capillary GC/ECD method allows the analyst the option of using 0.25-0.32 mm ID capillary columns (narrow-bore) or 0.53 mm ID capillary columns (wide-bore). The use of narrow-bore (0.25-0.32 mm ID) columns is recommended when the analyst requires greater chromatographic resolution. Use of narrow-bore columns is suitable for relatively clean samples or for extracts that have been prepared with one or more of the clean-up options referenced in the method. Wide-bore columns (0.53 mm ID) are suitable for more complex environmental and waste matrices. This option is not used by AML.

9.4.2 Dual-column analysis - The dual-column/dual-detector approach involves the use of two 30-m fused-silica open-tubular columns of different polarities, thus different selectivities towards the target compounds. The columns are connected to an injection tee and ECD detectors. Dual column is employed at AML.

9.4.3 GC temperature programs and flow rates:

9.4.3.1 A copy of the GC operating conditions for the analysis of PCBs as Aroclors is included in Appendix 1. Use the conditions in this method as guidance and establish the GC temperature program and flow rate necessary to separate the analytes of interest. When determining PCBs as Aroclors, chromatographic conditions should be adjusted to give adequate separation of the characteristic peaks in each Aroclor.

9.4.3.2 The retention times for the various peaks are included in the initial calibration (ICAL) summary, which is included in Appendix 3. Once established, the same operating conditions must be used for the analysis of samples and standards.

9.5 Calibration

9.5.1 Prepare calibration standards as described in the Standards section. Refer to AML SOPs based on EPA SW-846 Method 8000 for proper calibration techniques for both initial calibration and calibration verification. The SW-846 methods should be used when a suitable AML SOP is unavailable during method development and/or implementation. The calibration standards must contain the internal standard at the same concentration as the sample extracts. <u>NOTE</u>: Because of the sensitivity of the electron capture detector, the injection port and column should always be cleaned prior to performing the initial calibration.

9.5.2 When PCBs are to be quantitatively determined as Aroclors, the initial calibration consists of two parts, described below:

9.5.2.1 The standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. Thus, such a standard may be used to demonstrate the linearity of the detector and that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample. Therefore, an initial five-point calibration is performed using the mixture of Aroclors 1016 and 1260.

9.5.2.2 Standards of the other five Aroclors are necessary for pattern recognition. These standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1016/1260 mixture has been used to describe the detector response. In principle, the standards for these five Aroclors should be analyzed before the analysis of any samples. In practice, most samples are free of the Aroclors and the most commonly identified PCB is Aroclor-1260, which is present in the initial calibration mixture. In practice, sample extracts may be analyzed quantitatively for Aroclor-1016 and 1260 and screened for the presence of other Aroclors. If any Aroclor other than 1016 and 1260 appears to be present, a single-point calibration may be performed to establish response factors and pattern for the five Aroclors. In this case, the sample extracts should be reanalyzed. When reanalysis is performed, the analyst, at his discretion may set up a five point initial calibration for the identified Aroclor instead of relying on a single point calibration.

9.5.2.3 In situations where only a few Aroclors are of interest for a specific project, the analyst may employ a five-point initial calibration of each of the Aroclors of interest (e.g., five standards of Aroclor 1232 if this Aroclor is of concern) and not use the 1016/1260 mixture described in this SOP.

9.5.3 Establish the GC operating conditions as described above. Optimize the instrumental conditions for resolution of the target compounds and sensitivity. A 2-uL injection of each calibration standard is recommended. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest. Once established, the same opearting conditions must be used for the analysis of samples and standards.

9.5.4 Record the peak area (or height) for each congener (not used at AML) or each characteristic Aroclor peak to be used for quantitation.

9.5.4.1 Choosing five peaks (minimum required is 3 per SW-846 Method 8082 and 8082A) was recommended in an earlier version of the AML SOP. However, using small peaks that make up <25% of the largest peak caused higher variabilities for the quantitation.

Choosing only three peaks for each Aroclor is ecommended unless five peaks are required to satisfy specific clients.

9.5.4.2 The chosen peaks must be characteristic of the Aroclor in question. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of chosen peaks should include at least one peak that is unique to that Aroclor. Use a total of six to ten peaks (three to five peaks for each Aroclor, 1016 and 1260), none of which should be found in both of these Aroclors.

9.5.4.3 This discussion applies both to the primary column and the confirmation column. See Appendix 3 for a summary of the initial calibration and the list of peaks used for Aroclor 1016 and 1260.

9.5.4.4 Late-eluting Aroclor peaks are generally the most stable in the environment.

9.5.5 When determining Aroclors by the internal standard procedure (used at AML), calculate the response factor (RF) for each peak in the calibration standards relative to the internal standard using the equation that follows.

$$RF = \frac{A_s \text{ x } C_{is}}{A_{is} \text{ x } C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.

A_{is} = Peak area (or height) of the internal standard.

C_s = Concentration of the analyte or surrogate, in µg/L.

C_{is} = Concentration of the internal standard, in µg/L.

9.5.6 When determining PCBs as Aroclors by the external standard technique (seldom used at AML), calculate the calibration factor (CF) for each characteristic Aroclor peak in each of the initial calibration standards using the equation below.

$$CF = \frac{\text{Peak Area (or Height) in the Standard}}{\text{Total Mass of the Standard Injected (in nanograms)}}$$

Three to five sets of calibration factors will be generated for the Aroclor-1016 and Aroclor-1260, each set consisting of the calibration factors for each of the peaks chosen for this mixture. The single standard for each of the other Aroclors will generate calibration factors for each of the selected peaks.

9.5.7 The response factors or calibration factors from the initial calibration are used to evaluate the linearity of the initial calibration. Calculate the mean response or calibration factor, the standard deviation, and the relative standard deviation (RSD) for each Aroclor peak.

9.5.7.1 The 8082 and 8082A methods do not provide detailed guidance for handling the initial calibration data other than making the reference to SW-846 Method 8000. These methods and the chromatographic data systems are intended for the analysis of single-component analytes. The following guidance is used at AML. The analyst should obtain additional guidance from the QA Director or the Operations Manger to handle specific requests from clients.

9.5.7.2 Ideally, the %RSD for each Aroclor peak should be less than 20% to assume linearity. If five peaks are chosen and the %RSD is greater than 20% for any of the five Aroclor peaks, the mean %RSD for all five peaks should be less than 20%. This criteria applies to each Aroclor (1016 and 1260) present in the ICAL as well as any other Aroclor that may be run at five different concentrations.

9.5.7.3 In this SOP, each peak is treated as a separte analyte, although they are not used alone to report sample quantities for the Aroclor. If linearity cannot be demonstrated for all five peaks, one of the following options may be used:

9.5.7.3.1 Use three or four strong, better-performing peaks rather than using all five peaks for quantitation.

9.5.7.3.2 Use least square regression methods to calculate sample concentrations. For regression methods, the instrument response is treated as the dependent variable (y) and the standard amount is the independent variable (x). When “Cs” is used as “x”, use “As*Cis/Ais” as “y”. When “Cs/Cis” is used as “x”, use “As/Ais” as “y”. Do not include origin (0,0) as an extra calibration point.

9.5.7.3.3 Weighted least squares method is preferred for calculating concentrations more accurately at lower levels. A linear least squares equation (y = a*x + b) is preferred over non-linear calibration equations (y = a*x2 + b*x + c). The coefficient of determination (COD), coefficient of variation (r2) or correlation coefficient (r) should be used to measure the goodness of fit. For regression models to be used for quantitative purposes, the COD, r2 or r must be greater than or equal to 0.99. A value of 1.00 indicates a perfect fit.

9.5.7.3.4 Corrective action may be required if the criteria for %RSD, r, r2, or COD are not met. In practice, it may be better to check and correct the instrument and/or

calibration standards for potential problems and reanalyze the ICAL standards to meet linearity criteria.

9.5.7.3.5 An initial calibration (ICAL) should be considered a single event process. Consult the QA office or check applicable EPA methods (e.g. SW-846 Method 8000C and 8081A) for additional guidance on appropriate corrective action.

9.5.7.3.6 Re-fitting of ICAL – The selected model for ICAL may be subjected to an additional re-fitting check to establish representativeness of the model for each compound in each ICAL standard. This is an optional procedure, which is recommended but not required since automated data processing procedures may not be able to accommodate it. Calculate the %D between the calculated amount (Cc) for each compound in each ICAL standard from the ICAL model and its expected value (Ce). Ideally, the %D ((Cc-Ce) * 100 /Ce) for each compound in ICAL standard should meet the QC criteria for %RSD. It is possible for one or more ICAL standards to have %D exceeding the %RSD criteria, while the %RSD for ICAL model is within QC limits. The re-fitting procedure may be used to identify ICAL standards that need re-analysis or dropped from the calculation of ICAL model when re-analysis was not performed.

9.5.7.3.7 Check and optimize instrument operating conditions including automated peak integration procedures for appropriateness in addressing the chromatographic needs of the specific compound with problems.

9.5.7.3.8 If the problem appears to be associated with just one of the ICAL standards, that one standard may be reanalyzed once immediately (must be within 8-hours) after the ICAL. Discard all the results from the original analysis of the standard in question and replace it with the results from re-analysis.

9.5.7.3.9 When additional calibration standards (six or seven instead of five) are analyzed, narrowing the range of concentrations to be used for determining the goodness of fit is an acceptable procedure. Five or more standards should remain for the calculation of RSD or linear regression for each compound after one or more points are removed from the ICAL. More points (6 for second order and 7 for third order) may be needed for non-linear regression. It is generally prohibited to remove data points from within a calibration range,

while still retaining the extreme ends of the calibration range. Do not discard a middle point unless it appears to have an obvious problem and six or more standards were analyzed for ICAL and re-analysis of the affected standard was not performed within the required time limits. Changes to the upper end of the calibration range will affect the need to dilute samples above the range. A change to the lower range is not recommended since it will affect the method quantitation limit (MQL) used to report data. The default MQLs, which are based on the lowest ICAL concentration are stored in the LIMS and project-specific changes are not practical.

9.5.7.3.10 The refitting procedures may lead to failed %Ds for upper level calibration standards for average response factor methods. The analyst may take alternate corrective actions such as the analysis of sample extracts under dilution for the affected compounds.

9.5.7.3.11 The refitting procedures may lead to failed %Ds for lower level calibration standards for regression methods. The analyst may take appropriate corrective action such as flagging the sample result as an estimated amount ("J" flag) at the lower level encountered in the sample.

9.5.7.3.12 The term "calibration verification" (CALV) is used in this SOP to describe initial verification (ICV) of the initial calibration (ICAL) using second-source standards and subsequent continuing verifications (CCVs) using same-source standards. The response factors calculated during CALV are not used for sample quantitation since CALV standards are not used as "continuing calibration" standards permitted in some of the EPA methods.

9.5.7.3.13 Initial Calibration Verification (ICV) - Analysis of a second source ICV standard is required before any samples can be analyzed for USACE projects. Since second source standards are not employed for the analysis of LCS/LCSD and MS/MSD samples, ICV analysis is applicable for all projects. Calculate the percent difference (%D) or %Drift for the observed response factor in ICV from the mean RF as described for CCV. The %D or %drift should not exceed ±15% for each target compound. Document calibration verification in instrument printouts as described for the CCV. Check the ICAL and ICV standards if it is not

achievable for all the target compounds without any exception. Use professional judgment for non-USACE projects and for compounds that are not contaminants of concern (COC) for USACE projects. The ICV may be used as the beginning CCV for sample analysis.

9.5.7.3.14 If the above criteria are met, the analysis for analytical samples can begin.

9.6 Retention time windows

9.6.1 Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for the identification of PCBs as Aroclors. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability.

9.6.2 The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that cannot be confirmed upon further analysis.

9.6.3 Establish retention time windows based on multiple injection of the calibration standards over several days. Establish mean, standard deviation (SD), ± 2*SD for warning limits, and ±3*SD control limits for retention time window for each peak. See Appnedix 2 for more details about this procedure. Use professional judgment to widen the window beyond the control limits, if necessary.

9.7 Gas chromatographic analysis of sample extracts

9.7.1 The same GC operating conditions used for the initial calibration must be employed for samples analyses. Verify calibration each 12-hour shift by injecting calibration verification standards prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than once every 12-hours and at the end of the analysis sequence. Client specification and good laboratory practice at AML require the analysis of CCVs after every 10 samples (after every 5 samples if dirty extracts are involved) to minimize the number of samples requiring re-injection when QC limits are exceeded. A beginning CCV is also required if sample analyses are not performed immediately after an ICV following an ICAL. An ending CCV is always required.

9.7.2 For Aroclor analyses, the calibration verification standard should be a mid-level mixture of Aroclor 1016 and Aroclor 1260. The calibration verification process does not *require* analysis of the other Aroclor standards used for pattern recognition, but the analyst may wish to include

a standard for one of these Aroclors after the 1016/1260 mixture used for calibration verification throughout the analytical sequence.

9.7.2.1 The response factor, RF_v (or calibration factor, CFv) for each analyte calculated from the continuing calibration verification standard must not exceed a difference of more than ±15% when compared to the mean response factor, RF_m (or calibration factor, CF) from the initial calibration curve.

$$\% \text{ Difference} = \frac{(RF_m - RF_v) * 100}{RF_m}$$

$$\% \text{ Difference} = \frac{\overline{CF} - CF_v}{\overline{CF}} \times 100$$

9.7.2.2 If this criterion is exceeded for any calibration factor or response factor, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis. Document calibration verification on instrument printouts as described in Appendix 4.

9.7.2.3 If routine maintenance does not return the instrument performance to meet the QC requirements based on the last initial calibration, then a new initial calibration should be performed.

9.7.3 Inject a 2-uL aliquot of the concentrated sample extract.

9.7.4 Qualitative identifications of target analytes are made by examination of the sample chromatograms, as described in the following sections.

9.7.5 Quantitative results are determined for each identified analyte (Aroclor), using the procedures described in the following sections for either the internal or the external calibration procedure. If the responses in the sample chromatogram exceed the calibration range of the system, dilute the extract and reanalyze. Peak height measurements are recommended over peak area when overlapping peaks cause errors in area integration.

9.7.6 Each sample analysis must be bracketed with an acceptable initial calibration, calibration verification standard(s) (each 12-hour shift), or calibration standards interspersed within the samples. When a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that last met the QC criteria must be re-injected.

9.7.7 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements. It is *recommended (and used at AML)* that standards be

analyzed after every 10 samples (*required* after every 20 samples and at the end of a set) to minimize the number of samples that must be re-injected when the standards fail the QC limits. The sequence ends when the set of samples has been injected or when qualitative or quantitative QC criteria are exceeded.

9.7.8 Use the calibration standards analyzed during the sequence to evaluate retention time stability. If any of the standards fall outside their daily retention time windows, the system is out of control. Determine the cause of the problem and correct it.

9.7.9 If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract or replacement of the capillary column or detector is warranted. Rerun the sample on another instrument to determine if the problem results from analytical hardware or the sample matrix.

9.7 Qualitative identification

The identification of PCBs as either Aroclors or congeners using this method with an electron capture detector is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes. Tentative identification of an analyte occurs when a peak from a sample extract falls within the established retention time window for a specific target analyte. Each tentative identification must be confirmed using a second GC column of dissimilar stationary phase (as in the dual-column analysis), based on a clearly identifiable Aroclor pattern. The Aroclor pattern for the seven Aroclors described in this SOP are included in Appendix 4.

9.7.1 When simultaneous analyses are performed from a single injection, it is not practical to designate one column as the analytical (primary) column and the other as the confirmation column. Since the calibration standards are analyzed on both columns, the results for both columns must meet the calibration acceptance criteria. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.

9.7.2 When samples are analyzed from a source known to contain specific Aroclors, the results from a single-column analysis may be confirmed on the basis of a clearly recognizable Aroclor pattern when specifically requested by the Client to support rapid turnaround and/or high sample throughput requirements. This approach should not be used for routine evaluation of site samples for PCBs. This approach should <u>not</u> be attempted for samples from unknown or unfamiliar sources or for samples that appear to contain mixtures of Aroclors. In order to employ this approach, the analyst must document the following: (1) The peaks that were evaluated when comparing the sample chromatogram and the Aroclor standard. (2) The absence of major peaks representing any other Aroclor. (3) The source-specific information indicating that Aroclors are

anticipated in the sample (e.g., historical data, generator knowledge, etc.). (4) This information should either be provided to the data user or maintained by the laboratory.

9.7.3 GC/MS confirmation may be performed if the concentration of the Aroclors are atleast 100 times the lowest point on the calibration curve for SVOCs (See AML SOP based on EPA SW-846 Method 8270C).

9.8 Quantitation of PCBs as congeners.

The quantitation of PCB residues as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample.

9.8.1 Use the individual Aroclor standards (not the 1016/1260 mixtures) to determine the pattern of peaks on Aroclors 1221, 1232, 1242, 1248, and 1254. The patterns for Aroclors 1016 and 1260 will be evident in the mixed calibration standards. See Appendix 4 for a library of the Aroclor patterns.

9.8.2 Once the Aroclor pattern has been identified, compare the responses of 5 major peaks in the single-point calibration standard for that Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the mean individual response factor (calibration factor for external standard) for each of the 5 characteristic peaks chosen for Aroclor 1016 and 1260. If five point calibrations are run using any of the other Aroclors, use the mean RF for each peak. If multi-point calibrations are not performed, determine the concentration of the Aroclor using the RF established based on the single point. Perform this procedure for each Aroclor peak. A concentration is determined using each of the characteristic peaks and then those 5 concentrations are averaged to determine the concentration of that Aroclor.

9.8.3 Repeat the procedure to calculate Aroclor concentrations for both chromatographic columns. Calculate the percent RPD of the two concentrations and compare it to the EPA SW-846 Method 8000 criteria.

9.8.3.1 If the RPD is less than 40%, report the value from the primary GC column without qualifications, if no visible interference is present for one or more peaks in one of the columns.

9.8.3.2 If the %RPD is greater than 40%, report the value from the primary column as an estimated value ("J" flag) and document this problem on the case narrative. Use professional judgment for selecting value to report, if interference is suspected for the primary column. If significant interference that provides positive bias is present for the primary column, report data from the confirmation column for the affected analyte and document it in the case narrative.

9.8.4 Weathering of PCBs in the environment and changes resulting from waste treatment processes may alter the PCBs to the point that the pattern of a specific Aroclor is no longer recognizable. Samples containing more than one Aroclor present similar problems.

9.9 GC/MS confirmation may be used in conjunction with either single-or dual-column analysis if the concentration is sufficient for detection by GC/MS. Full-scan quadrupole GC/MS will normally require a higher concentration of the analyte of interest than selected ion monitoring techniques. The concentrations will be instrument-dependent, but values for full-scan quadrupole GC/MS may be as high as 500-ng/µL in the final extract since Aroclors consist of a number of compounds, whose total add up to the concentration described. The GC/MS must be calibrated for the specific target analytes. When using SIM techniques, the ions and retention times should be characteristic of the Aroclors to be confirmed. GC/MS confirmation should be accomplished by analyzing the same extract used for GC/ECD analysis and the extract of the associated blank. The base/neutral/acid extract (if also run on the same sample) and the associated blank may be used for GC/MS confirmation if the surrogates and internal standards do not interfere. However, if the compounds are *not* detected in the base/neutral/acid extract, then GC/MS analysis of the pesticide extract should be performed.

9.10 Chromatographic System Maintenance as Corrective Action - When system performance does not meet the established QC requirements, corrective action is required, and may include one or more of the following.

9.10.1 Splitter connections- For dual columns which are connected using a press-fit Y-shaped glass splitter or a Y-shaped fused-silica connector, clean and deactivate the splitter port insert or replace with a cleaned and deactivated splitter. Break off the first few inches (up to one foot) of the injection port side of the column. Remove the columns and solvent backflush according to the manufacturer's instructions. If these procedures fail to eliminate the degradation problem, it may be necessary to deactivate the metal injector body and/or replace the columns.

9.10.2 Metal injector body - Turn off the oven and remove the analytical columns when the oven has cooled. Remove the glass injection port insert (instruments with on-column injection). Lower the injection port temperature to room temperature. Inspect the injection port and remove any noticeable foreign material.

9.10.2.1 Place a beaker beneath the injector port inside the oven. Using a wash bottle, rinse the entire inside of the injector port with acetone and then rinse it with toluene, catching the rinsate in the beaker.

9.10.2.2 Consult the manufacturer's instructions regarding deactivating the injector port body. Glass injection port liners may require deactivation with a silanizing solution containing dimethyldichlorosilane.

9.10.3 Column rinsing - The column should be rinsed with several column volumes of an appropriate solvent. Both polar and nonpolar solvents are recommended. Depending on the nature of the sample residues expected, the first rinse might be water, followed by methanol and acetone. Methylene chloride is a good final rinse and in some cases may be the only solvent

required. The column should then be filled with methylene chloride and allowed to stand flooded overnight to allow materials within the stationary phase to migrate into the solvent. The column is then flushed with fresh methylene chloride, drained, and dried at room temperature with a stream of ultrapure nitrogen.

10.0 QUALITY CONTROL

10.1 Quality control procedures are used to ensure that proper sample preparation techniques, extract cleanup procedures, and instrument calibration procedures are performed. This involves the analysis of method blanks (MB), laboratory control samples (LCS), and matrix spikes (MS).

10.2 The frequency of analysis of LCS analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples. LCS can be run in duplicate to obtain precision (RPD) information. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. Document LCS results in Form-1 equivalents and recoveries in Form-3 Equivalents (See Appendix 8 for more details).

10.3 The frequency of analysis of MS analysis is equivalent to a minimum of 2 per 20 samples or 2 per batch if less than 20 samples. MS is run in duplicate to obtain precision (RPD) information. Document MS results in Form-1 equivalents and recoveries/RPDs in Form-3 Equivalents (See Appendix 7 for more details).

10.4 The frequency of analysis of MB analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples.

10.5 Include a calibration standard after each group of 20 samples (it is *recommended* that a calibration standard be included after every 10 samples to minimize the number of repeat injections) in the analysis sequence as a calibration check. The response factors for the calibration should be within 15 percent of the initial calibration. When this continuing calibration is out of this acceptance window, the laboratory should stop analyses and take corrective action.

10.6 Whenever quantitation is accomplished using an internal standard, internal standards must be evaluated for acceptance. The measured area of the internal standard must be no more than 50 percent different (-50% to +100%) from the area obtained during initial calibration. When the internal standard peak area is outside the limit, all samples that fall outside the QC criteria must be reanalyzed.

10.7 Sample Quality Control for Preparation and Analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). This includes the

addition of surrogates to each field sample and QC sample and evaluating their recoveries against QC limits. The surrogate recoveries are documented using Form-2 equivalents (See Appendix 9 for more details).

10.8 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair, spiked with the Aroclor 1016/1260 mixture. However, when specific Aroclors are known to be present or expected in samples, the specific Aroclors should be used for spiking. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. Since this knowledge may not always be available, AML will always analyze the MS in duplicate (MS and MSD). Sample duplicate analyses are not performed.

10.9 Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

11.0 **DATA VERIFICATION AND VALIDATION -** This section will provide guidance and procedures to assess if data produced using this SOP is valid and legally defensible. Document review and comments on the checklists (See Appendix 5). Verify that sample preparation logs are complete and the instrument run logs are available for review (See Appendix 11). Anomalies and exceptions should be documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on the checklists. Army (USACE) and Air Force (AFCEE) projects may have special requirements (See Appendix 13) that will require extra attention. A simple, universal procedure for evaluating and choosing appropriate corrective action doe not exist to deal with situations when one or more QC parameters are outside the acceptance window. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints. To make the matters simple, AML has adopted the QC limits provided in the DoD QSM as default limits for the surrogate, LCS, and MS recoveries.

11.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) projects may have additional requirements that are related to USACE laboratory validation (CX), USACE District requirements, government contract requirements, specific USACE project requirements or USACE Architect/Engineer (A/E) requirements. USACE-CX requirements (Summary of Method Quality Objectives for Method 8082) are included in Appendix 13.

11.1.1 The USACE requirements shown in Appendix 13 and elsewhere in this SOP are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the

legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may or may not be attainable for routine sample analysis. Since USACE MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

11.1.2 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality objectives and/or other QC requirements. Exceptions and data qualifications necessary should be documented on the checklists and eventually on case narratives.

11.1.3 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist, perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete. For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete. For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director will review the checklists (initial/date as necessary), prepare case narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

11.1.4 The blank criteria shown in Appendix 13 has been modified by USACE auditor(s) as one half of the applicable PQL (personal communications, USACE auditor, 1999).

11.2 HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

11.2.1 WATER - The holding times for PCBs is 7 days from sampling.

11.2.2 SOIL – A 14 day holding is used for soil and waste samples.

11.2.3 Holding times are established by comparing the chain of custody sampling date with the date of the injection after purging.

11.2.4 ACTION – If holding times have been exceeded, contact the client. All positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If

the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or innvalid (I).

11.3 INITIAL CALIBRATION – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the GC/ECD is capable of producing data within a range that is linear. Verification of the ICAL with a second source standard (ICV) is required for USACE and AFCEE projects (See Appendix 13). The ICV is performed and assessed similar to CCV (Section 11.4) except for special USACE and AFCEE requirements.

11.3.1 Compounds – Check to see if any Aroclor peak is outside the criteria of 20% or less for %RSD. If an Aroclor peak is outside the 20% criteria, check to see whether the mean %RSD for the three or five Aroclor peaks is within the 20% criteria.

11.3.2 ACTION - Use regression procedures for peaks not meeting the criteria, if the mean %RSD is within criteria. The "r" , “r_2” or “COD”, the measure of the fit for regression procedures should be >0.99. Apply criteria to each Aroclor and to data from both columns.

11.3.3 ACTION – Check and correct the standards, including the range of concentrations for appropriateness and repeat the ICAL analysis.

11.3.4 ACTION – Use a lesser number of peaks (minimum 3) if the criteria are not met for five peaks (desirable) selected for each Aroclor.

11.3.5 ACTION - Report the problem to the Project Manager and report detects and non-detects with aapropriate qualifiers for rapid TAT projects. Mention the ICAL anomaly in the case narrative.

11.4 INITIAL CALIBRATION VERIFICATION – Calibration verification includes initial calibration verification (ICV) and continuing calibration verifications (CCVs). A second source ICV is used to check the integrity of the ICAL standards used for calibrating the instrument. The 12-hour analytical shift begins with the injection of the ICV (instead of a CCV) for sample analyses that are performed immediately after an ICAL.

11.4.1 Target Compounds - Check to see if %D for any of the target compounds is outside the USACE QC criteria (<±15%)

11.4.2 ACTION - Check the ICAL and ICV standards for integrity. Replace one or more of the working standards (ICV first and ICAL later) for trouble shooting. Correct the problem(s) and repeat ICV analysis. Use professional judgment for non-USACE projects.

11.4.3 ACTION - When ICAL and ICV are acceptable based on SW-846 criteria, positive results and non-detects for target compounds not meeting the ±15% %D may be reported without data qualifications for non-USACE projects provided a valid CCV was obtained immediately following the

CCV. The ICAL and/or ICV standards should be inspected and replaced, if necessary for compliance with this requirement for USACE projects.

11.5 CONTINUING CALIBRATION VERIFICATION – The continuing calibration verification (CCV) is used to check that the instrument is still operating under the linear range of the initial calibration and has remained stable every 12 hours.

11.5.1 IS area counts (use mean IS area from ICAL for the %D of IS area) must not vary by more than a factor of 2 (-50% or + 100%) from the associated ICAL.

11.5.2 Compounds – Check to see if the %D for any Aroclor peak is outside the criteria of 15% or less. Check to see that the mean %D for all the peaks is <15%.

11.5.3 The %D for Aroclors present in the sample – The %D for the CCVs for each Aroclor peak before and after the sample analysis should be within QC limits when internal standards are not used. The %D for the CCV before sample analysis should be within QC limits when internal stanadrds are used.

11.5.4 The %D for Aroclors not present in the sample – The %D for the CCVs before and after the sample analysis may be >(+15%). Corrective action is not required unless the %D is < (-15%).

11.5.5 ACTION – Correct the problem and rerun the calibration verification and all associated samples. Positive results for compounds not meeting the criteria may be qualified as estimated (J) by the user (client). Report non-detects without qualifiers and mention these outliners in the case narrative.

11.6 METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Sample data qualification to alert blank contamination is performed by the laboratory. Sample data qualification based on all the blank results (laboratory MB, equipment blank, and other field blanks) is performed by the user (client). The processing of blanks helps eliminate reporting of false positives in project reports.

11.6.1 Method blanks should be free of all target anaytes and other interfering non-target analytes. To be acceptable, blank results should not exceed one half of the applicable MQL for any compound.

11.6.2 Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples. If levels greater than one half the PQLs are present, inspect and correct the problem. Reanalyze all associated samples. Contact client if other criteria are applicable. The only exception may be quick turn around projects.

11.6.3 ACTION – All positive results are reported with a “B” code, if contaminants are found in the method blank.

11.6.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, then no action is taken.

11.6.5 ACTION – Any compound detected in the sample which were also detected in associated method blank at levels greater than one half the PQLs should be re-extracted unless other client criteria are applicable.

11.6.6 ACTION – If contaminants resulting in GC/ECD peak saturation are present, all affected compounds are qualified as invalid (I).

11.7 SURROGATE RECOVERIES – Surrogates or system monitoring compounds are different structurally, but similar chemically to classes of target compounds. These surrogate compounds are added just prior to extraction of the samples and the percent recoveries of the surrogates are calculated and evaluated. The SW-846 method and DoD QSM recommeds the use of Decachlorobiphenyl as the only surrogate for PCBs analysis. The AML procedure involves sharing of a sample extract for the analysis of organochlorine pesticides (8081) and PCBs (8082), which are method analytes for other EPA methods (508.1 and 608). Both DCB and tetrachloro-m-xylene (TCMX) are used as surrogates and the QC limits recommended for DCB and TCMX for Method 8081 are also extended to the PCBs analysis, although sample extracts undergo different cleanup methods for the two procedures.

11.7.1 Document surrogate recoveries in Form-2 equivalents (See Appendix 9 for detials). Verify that the surrogate recoveries for both surrogate compounds (DCB and TCMX) are within QC limits (DoD QSM) for both surrogates) Flag outliers with an asterisk (*).

11.7.2 ACTION – When surrogate recoveries are outside control limits the sample must be re-analyzed. Reanalysis may be performed under dilution if original analysis should indicate high levels of target and non-target analytes. Reextraction and cleanup will be necessary if reanalysis does not resolve the problem. If surrogate recoveries are still outside the limits, report both sets of data and described the problem in the case narrative.

11.8 LABORATORY CONTROL STANDARD (LCS) – A LCS analysis is used to evaluate method competence in the absence of matrix effects. The LCS should be from a second source standard. The LCS may provide a mechanism to check method efficiency for target compounds without the sample matrix as a factor. See project plan and other available information (e.g. Appendix 13) for specific client requirements.

11.8.1 The QC limits for LCS recoveries are shown in Tables 1 and 2.

11.8.2 Check the results for the LCS recoveries to see if the results are within the acceptable range. Flag outliers with an asterisk (*).

11.8.3 ACTION – If deviations are noticed, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements, which may be compound specific. Check with the project manager about such requirements. Professional judgment may be used by the user in

evaluating sample data qualification options in conjunction with other QC data for the project.

11.9 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike data are used to evaluate the precision and accuracy of the analytical method on project samples. See project plan and other available information (e.g. Appendix 13) for specific client requirements.

11.9.1 The QC limits for MS/MSD percent recoveries are shown in Tables 1 and 2. The QC limits for relative percent difference (RPD) between the MS and MSD results are also shown in these tables.

11.9.2 Check the results for the MS/MSD recoveries. Flag outliers with an asterisk (*).

11.9.3 Perform one manual calculation using raw data and extraction sheet information.

11.9.4 ACTION – No action is taken on MS/MSD recoveries data alone to qualify all associated samples. A general rule is to have at least 90% of the target analytes within the control limits. Since matrix interference could lead to lead to serious analytical problems, the supervisor should be consulted when significant outliers are noted. Document QC outliers for %recovery and RPD on the checklist and on client reports (*) and draw attention to the QC outliers on the case narrative. No action may be taken on MS/MSD recovery data alone either to reanalyze samples and/or to qualify associated samples either by the analyst or by the end user. Professional judgment may be used by the user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

11.10 INTERNAL STANDARD PERFORMANCE – Internal Standard (IS) performance criteria ensures that the GC sensitivity and response is stable during each run.

11.10.1 IS area counts (use mean IS area from ICAL for the %D of IS area) must not vary by more than a factor of 2 (-50% or + 100%) from the associated ICAL/Calibration Verification standard. The internal standard areas for the CCV should not differ by a factor of 2 (-50% or + 100%) from the initial calibration of the same standard.

11.10.2 The retention time of the internal standard must not vary by more than 30 seconds from the associated daily calibration standard.

11.10.3 ACTION – If an internal standard is outside of QC limits of the associated daily standard, then re-analysis is required or a dilution may be required if it appears that interference's occur from target and non-target analytes at high concentrations relative to the ICAL. Do not dilute the sample to the point where all target analytes are non-detect. Professional judgement must be used when dilutions are required for IS criteria.

11.10.4 ACTION – If an internal standard is outside of QC limits of the associated ICAL or CCV standard, correct the problem and re-analyze samples with a valid CCV. If re-analysis has been performed and it is still out, it is of the professional judgement of the analyst to dilute or not to dilute the sample further so as to not to dilute out any target analytes.

11.10.5 ACTION - Report results without qualification describing the QC problem in the case narrative. The user (client) may qualify positive results for compounds quantitated using the affected IS as estimated (J). If a very low IS area is reported, indicating a loss of sensitivity, non-detects may require qualification as invalid (I) or rejected (R). Document recommendation for the client in the case narrative.

11.10.6 ACTION – If an IS retention time varies by more than +/- 30 seconds, the total ion profile must be checked for any false positives or negatives. If a large shift has occurred, then professional judgement must be used as to whether reanalyze or not. See previous section for guidance.

11.11 TARGET COMPOUND IDENTIFICATION – Criteria for GC/ECD qualitative analysis is to minimize the number of erroneous positive or negative identifications of compounds.

11.11.1 Professional judgement must be used when applying the criteria for GC/ECD analysis of target analytes. Take into account factors such as matrix effect and column bleed.

11.11.2 Positive compounds must be within +/- 0.06 relative time (RRT) units of the standard RRT. The results should be >MDL or other threshold for reporting positives (e.g. LLR, RL) on both columns.

11.11.3 The RPD for sample amounts from the two columns for identified compounds should be within <40%. Calculate RPD using Form-10 equivalents (See Appendix 6 for more details).

11.11.4 ACTION - If the RPD for positive results are <40%, report the result from the primary column without any flag. Report results greater than the upper limit of calibration with as estimated with an "E" flag.

11.11.5 ACTION - If the RPD is >40%, report the result from the primary column with a “J” flag. The result from the confirmation column may be reported with a “J” flag at the discretion of the analyst, if intererence is suspected for the compound of interest in the primary column.

11.11.6 ACTION - Samples with E coded data will require reanalysis with dilution. Both sets of results may need to be reported for some clients. See Table 1 and 2 for a summary of the MDLs, LLRs and MQLs.

11.11.7 ACTION - Adjust sample results and associated MDL, LLR, and MQL, for percent moisture and non-standard sample weights

11.11.8 ACTION - Report sample data in Form-1 equivalents (Appendix 12).

12.0 REFERENCES

12.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8081A, Organochlorine Pesticides by Gas Chromatography, December 1996.

12.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8082, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, December 1996.

12.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8082A, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Draft Revision 1A, May 1999.

12.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.

12.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

12.6 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3500B, Organic Extraction and Sample Preparation, December 1996.

12.7 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3520C, Continuous Liquid-Liquid Extraction, December 1996.

12.8 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3510C, Separatory Funnel Liquid-Liquid Extraction, December 1996.

12.9 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3511, Organic compounds in water by microextraction, Revision 0, November 2002.

12.10 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3540C, Soxhlet Extraction, December 1996.

12.11 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3541, Automated Soxhlet Extraction, December 1996.

12.12 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3545, Pressurized Fluid Extraction, December 1996.

12.13 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3550B, Ultrasonic Extraction, December 1996.

12.14 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3570, Microscale Solvent Extraction (MSE), Revision 0, November 2002.

12.15 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3600C, Cleanup, December 1996.

12.16 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3640A, Gel-Permeation Cleanup, December 1996.
12.17 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3660B, Sulfur Cleanup, December 1996.
12.18 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3665A, Sulfuric Acid/Permanganate Cleanup, December 1996.
12.19 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3580A, Waste Dilution, December 1996.
12.20 Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, EPA, 40 CFR Part 136, Method 608: Chlorinated Pesticides and PCBs, October 1984.
12.21 Methods for the Determination of Organic Compounds in Drinking Water, Supplement II, EPA/600/4-88-039, Method 508, Determination of Chlorinated Pesticides in Water by Gas Chromatography with an Electron Capture Detector, July 1991.
12.22 Methods for the Determination of Organic Compounds in Drinking Water, Method 508.1, Determination of Chlorinated Pesticides in Water by Gas Chromatography with an Electron Capture Detector.
12.23 USEPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-media, Multi-concentration, OLM02.0.
12.24 USEPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-media, Multi-concentration, OLM03.0.
12.25 USEPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-media, Multi-concentration, OLM04.0.
12.26 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.
12.27 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.
12.28 AML Standard Operating Procedures, Q-Water-RC.
12.29 AML Standard Operating Procedures, Q-WasteMgmt-RD.
12.30 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
12.31 AML Standard Operating Procedures, Q-Controlcharts-RC.

TABLE 1
Aqeous MDLs, LLRs, MQL and QC Limits for LCS/LCSD and MS/MSD Recoveries and RPDs Surrogate Recovery Limits

Polychlorinted Biphenyl (PCBs) Aroclors Analysis by GC/ECD

Analytical Management Laboratories

Preparation Method:	3510C													
Analysis Method:	8082													
	MDL	**MDL**	**MDL**	**MDL**	**LLR**	**MQL**	**Ratio (RQ)**		**DoD QSM LCS & MS Control Limits**		**DoD QSM LCS & MS "ME" Limits**			**RPD**
Compound	Spike	DB-5	DB-1701	Report	Report	Report	QL/DL	Flag	Lower	Upper	Lower	Upper	Limit	
	ug/L	ug/L	Ug/L	ug/L	ug/L	ug/L			**%R**	**%R**	**%R**	**%R**	%	
Aroclor-1016	1.0	0.13	0.27	**0.27**	0.5	**2.0**	7.5		25	145	25	145	50	
Aroclor-1260	1.0	0.45	0.55	**0.55**	0.5	**2.0**	3.6		30	145	30	145	50	
Other Aroclors:									Default AML Control Limits		Default AML "ME" Limits			
Aroclor – 1221	1.0	0.29	0.41	**0.41**	0.5	**2.0**	4.9		25	145	25	145	50	
Aroclor – 1232	1.0	0.29	0.41	**0.41**	0.5	**2.0**	4.9		25	145	25	145	50	
Aroclor – 1242	1.0	0.29	0.41	**0.41**	0.5	**2.0**	4.9		30	145	30	145	50	
Aroclor – 1248	1.0	0.29	0.41	**0.41**	0.5	**2.0**	4.9		30	145	30	145	50	
Aroclor – 1254	1.0	0.29	0.41	**0.41**	0.5	**2.0**	4.9		30	145	30	145	50	
Surrogates: (same as 8081)														
DCB									30	135	30	135		
TCMX									25	140	25	140		

Criteria for the ratio (RD) of mean amount found to MDL: >1 but <5 and for MQL/MDL ratios (RQ) that are less than 3.

QC limit for RPD is from the USACE document, Appendix I, EM 200-1-3, February 1, 2001.

LCS (extended to the MS) %R limits are from the DoD QSM, Final Revision 2, June 2002.

The surrogate recovery limits shown for DCB and TCMX are from DoD QSM limits for 8081.

The following criteria will apply for marginal exceedances:

Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).

NOTE: Marginal exceedance concept is not applicable to PCBS analysis. Control limits and ME limits are identical.

NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.

TABLE 2

Soil/Sediment MDLs, LLRs, MQL and QC Limits for LCS/LCSD and MS/MSD Recoveries and RPDs Surrogate Recovery Limits

Polychlorinted Biphenyl (PCBs) Aroclors Analysis by GC/ECD

Analytical Management Laboratories

Preparation Method:	3550C		MDL Ext. Date:		6/20/01					Analyst:		RRH	
Analysis Method:	8082		MDL Anal. Date:		6/23/01					Matrix:		Soil	
	MDL	**MDL**	**MDL**	**MDL**	**LLR**	**MQL**	**Ratio (RQ)**			**LCS Limits**		**MS Limits**	**RPD**
Compound	Spike	DB-5	DB-1701	Report	Report	Report	QL/DL	Flag	Lower	Upper	Lower	Upper	Limit
	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg			**%R**	**%R**	**%R**	**%R**	%
Aroclor-1016	10	0.81	1.21	**1.21**	5	**33.3**	27.5		50	130	40	140	50
Aroclor-1260	10	1.00	1.86	**1.86**	5	**33.3**	17.9		50	130	40	140	50
Other Aroclors:													
(calculated results)													
Aroclor - 1221	10	0.91	1.54	**1.54**	5	**33.3**	21.7		50	130	40	140	50
Aroclor - 1232	10	0.91	1.54	**1.54**	5	**33.3**	21.7		50	130	40	140	50
Aroclor - 1242	10	0.91	1.54	**1.54**	5	**33.3**	21.7		50	130	40	140	50
Aroclor - 1248	10	0.91	1.54	**1.54**	5	**33.3**	21.7		50	130	40	140	50
Aroclor - 1254	10	0.91	1.54	**1.54**	5	**33.3**	21.7		50	130	40	140	50
Surrogates (same as 8081):													
DCB									55	130	55	130	
TCMX									70	125	70	125	

Criteria for the ratio (RD) of mean amount found to MDL: >1 but <10 for soil and for MQL/MDL ratios (RQ) that are less than 3.

QC limit for RPD is from the USACE document, Appendix I, EM 200-1-3, February 1, 2001.

LCS (extended to the MS) %R limits are from the DoD QSM, Final Revision 2, June 2002.

The surrogate recovery limits shown for DCB and TCMX are from DoD QSM limits for 8081.

The following criteria will apply for marginal exceedances:

Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).

NOTE: Marginal exceedance concept is not applicable to PCBS analysis. Control limits and ME limits are identical.

NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.

Table 3
List of Appendices

Appendix Number	Appendix Topic
1	Gas Chromatography Conditions
2	Retention Time Window Summary
3	Initial Calibration (ICAL) Summary (Form-6 equivalents) Instrument Printouts
4	Calibration Verification (ICV and CCV) Summary (Form-7 equivalents) Standard Chromatograms – Aroclor Patterns Instrument Printouts
5	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
6	Sample Result Worksheets (Form-10 equivalents)
7	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
8	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
9	Surrogate Recovery Reports (Form-2 equivalents)
10	Method Detection Limit (MDL) Study Summary
11	QC Association Forms (Form-4 equivalents) Sample Preparation Logsheets Instrument Run Log
12	Sample Reports (Form-1 equivalents)
13	USACE Method Quality Objectives for Method 8082 AFCEE MQOs for Method 8082 DoD QSM MQOs and Limits for percent recoveries

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices

STANDARD OPERATING PROCEDURES

No. O-PCBs-RD

POLYCHLORINATED BIPHENYLS BY GC/ECD (EPA 508.1, 608 and SW846-8082A)

February 24, 2006

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure, which is based on EPA methods (508.1, 608, and SW-846 Method 8082 and 8082A) is used to determine the concentrations of polychlorinated biphenyls (PCBs) as Aroclors in extracts from solid and aqueous matrices. Open-tubular, capillary columns are employed with electron capture detectors (ECD). When compared to packed columns, these fused-silica, open-tubular columns offer improved resolution, better selectivity, increased sensitivity, and faster analysis. The target compounds listed below may be determined by either a single- or dual-column analysis system.

Compound	CAS Registry No.
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5

1.2 Aroclors are multi-component mixtures. When samples contain more than one Aroclor, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of Aroclors that have been subjected to environmental degradation ("weathering") or degradation by treatment technologies. Such weathered multi-component mixtures may have significant differences in peak patterns than those of Aroclor standards.

1.3 Quantitation of PCBs as Aroclors is appropriate for many regulatory compliance determinations, but is particularly difficult when the Aroclors have been weathered by long exposure in the environment.

1.4 Another approach based on PCB congeners may afford greater quantitative accuracy when PCBs are known to be present and it is of particular value in determining weathered Aroclors. However, the congener method may not be useful when regulatory requirements are based on Aroclor concentrations. The laboratory does not employ the congener approach for the analysis of PCBs.

1.5 Compound identification based on single-column analysis should be confirmed on a second column, or should be supported by at least one other qualitative technique. This SOP describes analytical conditions for a second gas chromatographic column that can be used to

confirm the measurements made with the primary column. GC/MS (SW-846 Method 8270) can also be used as a confirmation technique when sensitivity permits.

1.6 This SOP describes the dual-column option, allowed by the method. The option allows a hardware configuration of two analytical columns joined to a single injection port. The option allows one injection to be used for dual-column analysis. Analysts are cautioned that the dual-column option may not be appropriate when the instrument is subject to mechanical stress, many samples are to be run in a short period, or when highly contaminated samples are analyzed.

1.7 The analyst must select columns, detectors and calibration procedures most appropriate for the specific analytes of interest in a study. Matrix-specific performance data must be established and the stability of the analytical system and instrument calibration must be established for each analytical matrix (e.g., hexane solutions from sample extractions, diluted oil samples, etc.). Example chromatograms and GC conditions are provided as guidance.

1.8 The MDLs for Aroclors vary in the range of 0.054 to 0.90 µg/L in water and 57 to 70 µg/kg in soils. MDLs and MQLs for the laboratory are shown in Tables 1 and 2.

1.9 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs (GC) and skilled in the interpretation of gas chromatograms.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample (approximately 1 L for liquids, 2-g to 30-g for solids) is extracted using the appropriate matrix-specific sample extraction technique.

2.2 Aqueous samples are extracted at neutral pH with methylene chloride using Method 3510 (separatory funnel), Method 3520 (continuous liquid-liquid extractor), Method 3535 (solid-phase extraction) or other appropriate technique.

2.3 Solid samples are extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) using AML SOPs based on EPA SW-846 Method 3540 (Soxhlet), Method 3541 (automated Soxhlet), Method 3550 (sonication) or other appropriate technique. The SW-846 methods should be used when a suitable AML SOP is unavailable during method development and/or implementation.

2.4 Tissue samples may be extracted using Method 3562 (supercritical fluid extraction) or other techniques such as those described in Section 2.3 for solid samples.

2.5 Extracts for PCB analysis may be subjected to a sulfuric acid cleanup or a sulfuric acid/potassium permanganate cleanup (AML SOP based on EPA SW-846 Method 3665) designed specifically for these analytes. The SW-846 methods should be used when a suitable AML SOP is unavailable during method development and/or implementation. A summary of the sulfuric acid cleanup procedure used by AML is included in this SOP. This cleanup technique will remove (destroy) many single component organochlorine or organophosphorus pesticides. Therefore, these extracts

may not be used for the analysis of organochlorine pesticide compounds, if these cleanups are performed.

2.5 After cleanup, the extract is analyzed by injecting a 2-uL aliquot into a gas chromatograph with a narrow- or wide-bore fused silica capillary column and electron capture detector (GC/ECD).

2.6 The chromatographic data is used to determine the seven Aroclors listed in Section 1.1.

3.0 INTERFERENCES

3.1 Refer to AML SOPs based on SW-846 Methods 3500, 3600, and 8000 for a discussion of interferences. The SW-846 methods should be used when a suitable AML SOP is unavailable during method development and/or implementation. A summary of these procedures is included in this SOP.

3.2 Interferences co-extracted from the samples will vary considerably from matrix to matrix. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation. Sources of interference in this method can be grouped into three broad categories.

3.2.1 Contaminated solvents, reagents, or sample processing hardware.

3.2.2 Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.

3.2.3 Compounds extracted from the sample matrix to which the detector will respond (e.g. chlorinated pesticdes and herbicides).

3.3 Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determinations.

3.3.1 Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination.

3.3.2 Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

3.3.3 These materials can be removed through the use of AML SOP based on EPA SW-846 Method 3665 (sulfuric acid/permanganate cleanup). The SW-846 methods should

be used when a suitable AML SOP is unavailable during method development and/or implementation. A summary of these procedures is included in this SOP.

3.4 Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water. Drain the glassware, and dry it in an oven at 130°C for several hours, or rinse with methanol and drain. Store dry glassware in a clean environment.

NOTE: Oven-drying of glassware used for PCB analysis can increase contamination because PCBs are readily volatilized in the oven and spread to other glassware. Therefore, exercise caution, and do not dry glassware from samples containing high concentrations of PCBs with glassware that may be used for trace analyses.

3.5 Elemental sulfur (S_8) is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. Sulfur can be removed through the use of AML SOP based on EPA SW-846 Method 3660. The SW-846 methods should be used when a suitable AML SOP is unavailable during method development and/or implementation.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph - An analytical system complete with gas chromatograph suitable for on-column and split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and recorder/integrator or data system: Two Hewlett Packard (Agilent) Model 5890 Series II equipped with dual ECD detectors, and two Agilent autosamplers (Model 7673/7673A).

4.2 GC columns - A dual-column approach is used, which involves a single injection that is split between two columns that are mounted in a single injector in a single gas chromatograph. This dual-column approach employed at AML consists of two 0.25 mm ID columns.

4.2.1 Narrow-bore columns that are installed in split/splitless (Grob-type) injectors. Column pair is mounted in a press-fit Y-shaped glass 3-way union splitter (J&W Scientific, Catalog No. 705-0733) or a Y-shaped fused-silica connector (Restek, Catalog No. 20405), or equivalent.

4.2.1.1 30 m x 0.25 mm ID fused silica capillary column chemically bonded with SE-54 (DB-5 or equivalent), 1 µm film thickness.

4.2.1.2 30 m x 0.25 mm ID fused silica capillary column chemically bonded with 14% cyanopropylmethylpolysiloxane (DB-1701, or equivalent), 1.0 µm film thickness.

4.3 Column rinsing kit - Bonded-phase column rinse kit (J&W Scientific, Catalog No. 430-3000), or equivalent.

4.4 Volumetric flasks - 10-mL and 25-mL, for preparation of standards.

5.0 REAGENTS

5.1 Reagent grade or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate standards) at 4 degrees C in polytetrafluoroethylene (PTFE)-sealed containers in the dark. When a lot of standards is prepared, it is recommended that aliquots of that lot be stored in individual small vials. All stock standard solutions must be replaced after one year or sooner if routine QC indicates a problem. All other standard solutions must be replaced after six months or sooner if routine QC indicates a problem.

5.2 Sample extracts prepared by AML SOPs based on SW-846 Methods 3510, 3520, 3540, 3541, 3545, or 3550 need to undergo a solvent exchange step prior to analysis. The following solvents are necessary for dilution of sample extracts. All solvent lots should be pesticide quality or equivalent and should be determined to be phthalate-free.

5.2.1 n-Hexane, C_6H_{14}

5.2.2 Isooctane, $(CH_3)_3CCH_2CH(CH_3)_2$

5.3 The following solvents may be necessary for the preparation of standards. All solvent lots must be pesticide quality or equivalent and should be determined to be phthalate-free.

5.3.1 Acetone, $(CH_3)_2CO$

5.3.2 Toluene, $C_6H_5CH_3$

5.4 Organic-free reagent water.

5.5 Stock standard solutions - The stock standard solutions are purchased from Ultra Scientific as certified solutions. Second source standards are purchased from Supelco.

5.5.1 The surrogate 2,4,5,6-Tetrachloro-m-xylene (TCMX) is purchased from Ultra (cat # IST-440) at a concentration of 2,000-ug/mL.

5.5.2 The surrogate, decachlorobiphenyl is purchased from Ultra (cat # PPS-150) at a concentration of 1,000-ug/mL.

5.5.3 The surrogate spiking solution containing TCMX and DCB at a concentration of 200-ug/mL each is purchased from Ultra (Cat # ISM-320).

5.5.4 The Aroclor-1016/1260 mixture is purchased from Ultra (cat # PPM-8082) at a concentration of 1,000-ug/mL each in hexane:toluene (1:1).

5.5.5 Individual standards of the various Aroclors are also purchased at a concentration of 1,000-ug/ml in iso-octane:

5.5.5.1 Aroclor-1016, catalogue #EPA-1282
5.5.5.2 Aroclor-1221, catalogue # EPA-1292
5.5.5.3 Aroclor-1232, catalogue # EPA-1302
5.5.5.4 Aroclor-1242, catalogue # EPA-1312
5.5.5.5 Aroclor-1248, catalogue # EPA-1342
5.5.5.6 Aroclor-1254, catalogue # EPA-1352
5.5.5.7 Aroclor-1260, catalogue # EPA-1362
5.5.5.8 Aroclor-1262, catalogue # EPA-1372
5.5.5.9 Aroclor-1268, catalogue # EPA-1382

5.5.6 Second source Aroclor-1016/1260 (at 500-ug/mL each) mixture containing the two surrogates (at 50-ug/mL each) is purchasd from Supelco (46846-u) for the preparation of initial calibration verification (ICV) standards, which are used for compliance with USACE requirements.

5.5.7 Second source individual Aroclor solutions (1000-ug/mL in isooctane) are purchased from Supelco:

5.5.7.1 Aroclor-1016, catalogue #48097
5.5.7.2 Aroclor-1221, catalogue #48098
5.5.7.3 Aroclor-1232, catalogue #44805
5.5.7.4 Aroclor-1242, catalogue #44806
5.5.7.5 Aroclor-1248, catalogue #44807
5.5.7.6 Aroclor-1254, catalogue #44808
5.5.7.7 Aroclor-1260, catalogue #44809
5.5.7.8 Aroclor-1262, catalogue #44810

5.5.8 Internal Standard (IS) - The compound, 1-bromo-2-nitrobenzene is used as an internal standard, which is added to each sample extract prior to analysis, and included in each of the initial calibration standards. The stock standard solution is purchased through Ultra (cat # PPS-351) at a concentration 5000-ug/ml.

5.5.8.1 An intermediate IS solution is prepared by diluting 1.0-mL of the stock solution (5000-ug/mL) with 99-mL of iso-octane for a final concentration of 50-ug/mL or 50,000-ng/mL.

5.5.8.2 An aliquot (10-uL) of this solution is spiked 1.0-ml of sample extracts and calibration standards for a final IS concentration of 500-ng/mL in the solution injected into the gas chromatograph.

5.6 Calibration standards for Aroclors

5.6.1 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 and 1260 at five concentrations should be sufficient to demonstrate the linearity of the detector response without the necessity of performing initial calibrations for each of the seven Aroclors. In addition, such a mixture can be used as a standard to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample.

5.6.2 Default AR1016/1260 Multi-point Initial Calibration (ICAL) Standards - Using standards obtained from Ultra, prepare a minimum of five calibration standards containing the two surrogates (five different levels), the internal standard (one level) and equal concentrations of both Aroclor 1016 and Aroclor 1260 (five different levels).

5.6.2.1 Dilute 200-uL of the 1,000-ug/mL mixed Aroclor 1016/1260 standard (Ultra PPM-8082), 100-uL of the mixed surrogate standard (ISM-320) to 100-ml with acetone for a final concentration of 2000 ng/ml of each Aroclor and 200-ng/mL for each surrogate. This solution expires one year from the date of preparation. Replace sooner if signs of degradation is observed.

5.6.2.2 The 2000 ng/ml concentration standard (Section 5.6.2.1) is the high point of the calibration for the Aroclors. Add 10-uL of the IS intermediate solution (Section 5.5.8.1) to 1-mL of the 2000-ng/mL standard in a 1-mL GC injection vial.

5.6.2.3 From the 2000-ng/ml standard, 400-uls is diluted to 1.0 ml for a final concentration of 800-ng/ml. Add 10-uL of the IS intermediate solution (Section 5.5.8.1) to 1-mL of the 800-ng/mL standard in a 1-mL GC injection vial.

5.6.2.4 From the 2000-ng/ml standard, 300-uls is diluted to 1.0 mls for a final concentration of 600-ng/ml. Add 10-uL of the IS intermediate solution (Section 5.5.8.1) to 1-mL of the 600-ng/mL standard in a 1-mL GC injection vial.

5.6.2.5 From the 2000-ng/ml standard, 200-uls is diluted to 1.0 mls for a final concentration of 400-ng/ml. Add 10-uL of the IS intermediate solution (Section 5.5.8.1) to 1-mL of the 400-ng/mL standard in a 1-mL GC injection vial.

5.6.2.6 From the 2000-ng/ml standard, 100-uls is diluted to 1.0 mls for a final concentration of 200-ng/ml. Add 10-uL of the IS intermediate solution (Section 5.5.8.1) to 1-mL of the 200-ng/mL standard in a 1-mL GC injection vial.

5.6.2.7 The respective concentration of the surrogates in the five calibration standard solutions are as follows: 200-ng/mL, 80-ng/mL, 60-ng/mL, 40-

ng/mL, and 20-ng/mL. The internal standard concentration is 500-ng/mL in each of the ICAL standard solutions.

5.6.2.8 Injection vials containing the five point calibration standards are prepared daily.

5.6.3 Single standards of each of the other five Aroclors are required to aid the analyst in pattern recognition. Assuming that the Aroclor 1016/1260 standards described in Sec. 5.6.2 have been used to demonstrate the linearity of the detector, these single standards of the remaining five Aroclors are also used to determine the calibration factor for each Aroclor. Prepare a daily calibration standard for each of these Aroclors at a concentration, which should correspond to the mid-point of the linear range of the detector (1000 ng/ml).

5.6.3.1 Dilute 100-uL of the 1,000-ug/mL Aroclor standard from Ultra (See Section 5.5.5.2 to 5.5.5.6) and 100-uL of the mixed surrogate standard (ISM-320) to 100-ml with acetone for a final concentration of 1000 ng/ml of each Aroclor (present in individual solutions) and 200-ng/mL for each surrogate. The intermediate solution should be replaced six months from the date of preparation. Replace sooner if signs of degradation is observed.

5.6.3.2 Add 10-uL of the IS intermediate solution (Section 5.5.8.1) to 1-mL of the 1000-ng/mL standard in a 1-mL GC injection vial.

5.6.3.3 The working standards (Section 5.6.3.2 and 5.6.3.4) are prepared daily, whenever a new ICAL curve is generated.

5.6.3.4 Prepare individual single concentration standards of Aroclor-1262 (Section 5.5.5.8) and Aroclor-1268 (Section 5.5.5.9) for special projects as described in Section 5.6.3.1. These Aroclors are not standard target analytes.

5.6.4 Custom Aroclor Multi-point Initial Calibration (ICAL) Standards - For projects that require multi-point calibration with the Aroclor of concern, prepare a minimum of five calibration standard solution using the specified Aroclor instead of the AR1016/1260 mixture. The Aroclor 1016/1260 standards need not be used if a specific Aroclor is known to be present in site samples. Refer to the project specific Quality Assurance Project Plan for specific information.

5.6.4.1 For compliance with DoD requirements, prepare separate multi-point ICAL standards for each of the remaining five Aroclors (1221, 1232, 1242, 1248, and 1254) using stock standards (see Section 5.5.5) that are used to prepare default mid-level ICAL standards of these Aroclors for initial analysis.

5.6.4.2 IThe intermediate and working calibrations standards are prepared similiarly to 5.6.2.1 to 5.6.2.8. Aroclor stock standards described in Sections 5.5.5.1 to 5.5.5.9 are used.

5.6.5 Initial Calibration verification (ICV) - The ICV solution is prepared from a second source of stock standard solutions.

5.6.5.1 ICV for default ICAL - Prepare an ICV intermediate solution from the Supelco standard (See Section 5.5.6), which contains the two default ICAL target analytes (Aroclor-1016 and -1260) at a concentration of 500-ug/mL and the two surrogates at 50-ug/mL. Dilute 200-uL of the Supelco standard to 100-ml with acetone for a final concentration of 1000 ng/ml of each Aroclor and 100-ng/mL for each surrogate. This solution expires one year from the date of preparation. Replace sooner if signs of degradation is observed.

5.6.5.2 Add 10-uL of the IS intermediate solution (Section 5.5.8.1) to 1-mL of the 1000-ng/mL ICV intermediate standard in a 1-mL GC injection vial.

5.6.5.3 The working standard (Section 5.6.5.2) is prepared daily, whenever a new ICAL curve is generated.

5.6.5.4 Prepare second-source ICV standards for each of the remaining five Aroclors (1221, 1232, 1242, 1248, and 1254) using second-source stock standards (see Section 5.5.7).

5.7 Internal standard

5.7.1 The SW-846 recommeds the use of Decachlorobiphenyl (DCB) as the internal standard for PCB congener analysis. Although the use of an internal standard is not required for the analysis of Aroclors, AML uses the compound, 1-bromo-2-nitrobenzene as an internal standard (IS), to keep parity with other EPA determinative methods (e.g. 8081A and 8081B).

5.7.2 The internal standard is added to each sample extract prior to analysis, and included in each of the initial calibration standards. An aliquot (10-uL) of the IS solution is used to spike 1.0-mL of the sample extracts and calibration standards for a final concentration of 500-ng/ml in the solution injected into the GC.

5.8 Surrogate Sample Spiking Solutions

5.8.1 The SW-846 recommeds the use of Decachlorobiphenyl as the surrogate for PCBs analysis. The AML procedure involves sharing of a sample extract for the analysis of organochlorine pesticides (8081) and PCBs (8082), which are method analytes for other EPA methods (508.1 and 608). Both DCB and tetrachloro-m-xylene (TCMX) are used as surrogates, and they are added to each sample prior to extraction. The stock surrogate solution is purchased through Ultra Scientific (Catalog # ISM-321X) at a concentration of 200 ng/mL. Spike 1.0-ml of this solution to each sample including QC samples prior to extraction. The final extract (5-ml final volume) concentration will be 40-ng/mL.

5.9 LCS and MS Spike Standards

5.9.1 The LCS spiking solution is prepared from the source of stock standard solution (Ultra cat # PPM-8082) that is used for ICAL. Dilute 300-uL of this

solution to 100-mL with acetone. The resulting matrix spike solution is at a concentration of 3,000-ng/mL. Spike each LCS and LCSD sample with 1.0-mL of this 3000-ng/mL standard. The final extract (5-ml final volume) concentration will be 600-ng/mL in each of the two Aroclors (1016 and 1260).

5.9.2 The MS spiking solution is identical to the LCS spiking solution. Spike each MS and MSD sample with 1.0-mL of this 3000-ng/mL standard. The final extract (5-ml final volume) concentration will be 600-ng/mL in each of the two Aroclors (1016 and 1260).

5.9.3 Employ a similar procedure to prepare LCS and/or MS spiking solution for each of the remaining five Aroclors (1221, 1232, 1242, 1248, and 1254) using the same-source stock standards (see Section 5.5.5) described above. These solutions should also be used to demonstrate continuing analytical capabilities for each of the five Aroclors by analyzing a LCS pair at least annually.

6.0 SAMPLE PRESERVATION HANDLING & STORAGE

6.1 One litre amber glass bottles with Teflon lined screw caps may be used for collecting aqueous samples in accordance with EPA SW-846 method 8082. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 2 bottles for each water sample to be collected. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. See project specific field sampling plan (FSP) for sample collection and preservation instructions. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.

6.2 One 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples in accordance with EPA SW-846 Method 8082. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each solid sample. Quality control samples such as MS and MSD should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. These samples may be shared with samples collected for other analyses such as semivolatiles (8270C), chlorinated pesticides (8081A), diesel (DRO, 8015-mod.) and other pesticides (8140, etc.).

6.3 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction. Analytical data generated outside of the specified holding times must be considered to be minimum values only. The holding time for the extraction of aqueous samples is 7 days from the date of sample collection. The holding times for the extraction of soil, sediment and waste samples is 14 days from the date of sample collection. The holding time for the instrumental analysis of sample extracts is 40 days from the date of extraction.

7.0 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control contain a number of regulated compounds at high concentrations. The toxicity of these compounds present in such complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.
7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.
7.3 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.
7.4 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.
8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.
8.3 Aqueous and soil samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters after the solvent(s) have been allowed to evaporate in a hood.

9.0 **PROCEDURE**

9.1 Sample extraction – Aqueous Samples

9.1.1 Refer to specific AML extraction SOPs based on EPA SW-846 Method 3500B for guidance in choosing the appropriate extraction procedure. The SW-846 methods including the overview (Method 3500B) should be used when a suitable AML SOP is unavailable during method development and/or implementation. The microextraction procedures described in the SOP on organochlorine pesticides analysis may also be used for rapid TAT projects in support of remediation efforts. A summary of the SW-846 aqueous extraction methods and associated AML SOPs are included in this SOP.

9.1.2 Manual Liquid/Liquid Extraction (Method 3510C) - In general, water samples are extracted at a neutral pH with methylene chloride using a separatory funnel (AML SOP based on SW-846 Method 3510C) or a continuous liquid-liquid extractor (Method 3520C, not used at AML).

9.1.2.1 Using a black marker, mark the meniscus of the water in the sample container. Pour the entire contents of the container into a 2-liter separatory funnel. Rinse the sample container with 3 aliquots of 20 mls methylene chloride and place the rinse in the separatory funnel. Add distilled water to the sample container up to the marked meniscus. Pour the distilled water into a 1-liter graduated cylinder. Record this measured volume in the Sample Extraction Log Book. Using pH indicating paper (0-14) record the pH of the

sample (sample should be at a neatral pH). This is accomplished by dipping the tip of a Pasteur pipette into the water and placing the droplets of water on the pH paper. Never place the pH paper into the water sample. Add 1 mL of surrogate solution to all of the samples (including QC) and 1.0-ml of appropriate spiking solution to the LCS/LCSD & MS/MSD. Adjust, if necessary, the pH to 7 using either 50/50 sulfuric acid solution or 10N sodium hydroxide solution.

9.1.2.2 An alternate procedure, which may be employed to measure out water samples is described below. Using a 1-Liter graduated cylinder, measure out 1.00 liter (or lower volumes as low as 100-mL for high concentration samples) of the sample, and transfer it to a 2000-mL separatory funnel. Record the exact volume in the organic Extraction logbooks. This alternate procedure may be used to measure out lower sample volumes for the re-extraction of heavily contaminated samples with surrogate recoveries outside the QC limits, which are diluted out during analysis. This procedure may also be used to measure out smaller aliquots (e.g. 500-mL) of the sample for MS/MSD analysis, when additional sample containers were not collected for these analyses.

9.1.2.3 Add 60-mLs of methylene chloride to the separatory funnel and shake for 2 minutes. Take care to vent the separatory funnel to relieve excess pressure. Allow the organic layer to separate from the water layer for a minimum of 10 minutes. Drain the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus. Repeat the extraction 3 more times using 60 mLs of methylene chloride.

9.1.2.4 Add two boiling chips to the K-D flask and attach a three ball Snyder column. Prewet the Snyder column with 1 mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1-ml to 5-ml, add 5-ml of iso-octane or hexane and allow sample to come back down. Remove the K-D flask and allow cooling for at least 10 minutes.

9.1.2.5 Concentrate the extract by nitrogen blow-down to 1.0-mL making sure **all** methylene chloride has been removed. Adjust sample to 5.0-ml and proceed to analyze by GC/ECD. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 500-ng/mL. NOTE: When sample extracts prepared for organochlorine pesticides are used to screen for PCBs, the internal

standard concentration that is used is 50-ng/mL in the final extract and in calibration solutions.

9.2 Sample extraction – Soil Samples: Solid samples are extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) using one of the Soxhlet extraction (AML SOPs based on Method 3540C or 3541) procedures, ultrasonic extraction (AML SOP based on Method 3550B), or other appropriate procedure. Use of hexane-acetone for extraction, which is also used for the extraction of soil samples for organochlorine pesticides generally reduces the amount of interferences that are extracted and improves signal-to-noise. The microextraction procedures described in the SOP on organochlorine pesticides analysis may also be used for rapid TAT projects in support of remediation efforts. A summary of the SW-846 soil extraction methods and associated AML SOPs are included in this SOP.

9.2.1 Sample Extraction by Sonication – SW-846 Method 3550

9.2.1.1 Add 30-g of soil to a 500 mL beaker. Record the exact weight in the organic Extraction Log Book. Add enough sodium sulfate to dry the soil and mix well with a spoon. Add 1.0 mL of surrogate solution to all the samples (including QC) and 1.0-ml of appropriate spike spiking solutions to the LCS/LCSD & MS/MSD samples.

9.2.1.2 Add 50- to 100-mLs of the aceone:methylene chloride (20:80) mixture to the beaker and mix the contents with a spoon for 1 minute. Sonicate the soil/methylene chloride mixture for 3 minutes ensuring that the soil is free flowing and not in clumps. Decant the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus.

9.2.1.3 Extract the sample twice more with additional 50- to 100-mL aliquots of the solvent mixture. The solution should be sonicated for 3 minutes each time. Decant the solution in to the same K-D as described above.

9.2.1.4 Add two boiling chips to the K-D flask and attach a three ball Snyder column. Prewet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1-mL to 5-mL, add 5-mL of iso-octane or hexane and allow sample to come back down. Remove the K-D flask and allow to cool for at least 10 minutes.

9.2.1.5 Concentrate the extract by nitrogen blow-down to 1.0 mLs making sure **all** methylene chloride as been removed. Adjust sample to 5-ml and proceed to analyze by GC/ECD if no cleanup procedures are required. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 500-ng/µL. NOTE: When

sample extracts prepared for organochlorine pesticides are used to screen for PCBs, the inetrnal standard concentration that is used is 50-ng/mL in the final extract and in calibration solutions.

9.2.2 Sample Extraction by Automated Soxhlet Procedure A – SW-846 Method 3541

9.2.2.1 Automated Soxhlet Extraction Procedure-A involves IKA equipment, which are capable of handling 30-g sample aliquots. The set-up consists of six 4-position extractors to handle a total of 24 samples, which will include up to 5 QC samples (MB, LCS, LCSD, MS, and MSD). The six extractors, equipped with independent heating and cooling systems are controlled by a single personal computer. Follow manufacturer instructions for setting up and operating the IKA Soxhlet equipment.

9.2.2.2 Weight out 30-g of the soil sample to three significant numbers (±0.1-g) into 500-mL beakers. Record the exact weight in the organic Extraction logbook (See Appendix 11 for documentation). Add enough sodium sulfate (60-g) to dry the soil and mix well with a spoon. When the soil is dry (freely flowing powder), the sample is ready for Soxhlet extraction.

9.2.2.3 Clean and completely assemble the IKA equipment. Raise the condenser from the top vessel. Assemble the top vessel with the stainless steel filter base and a new disposable Teflon micron-sized filter paper. Transfer the soil/drier mixture quantitatively to the top vessel. Add 1.0-mL of surrogate solution to all the samples (including QC) and 0.25-mL of the appropriate spiking solutions to the LCS/LCSD & MS/MSD samples. Add 100-mL of the solvent mixture (methylene chloride:acetone 80:20) to the bottom vessel and tightly screw the top vessel on to the bottom vessel containing solvent. Replace the condenser unit on the top vessel. Turn the unit on and allow the unit to cycle (45-minutes). Completely drain the solvent from the sample chamber using the instrument cycle control mechanism. Repeat the extraction process twice using the same solvent contained in the bottom vessel. Decant the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus.

9.2.2.4 Add two boiling chips to the K-D flask and attach a three-ball Snyder column. Prewet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1-ml, add 5-mL of hexane and continue with solvent removal. When the extract reaches 1 to 2-mL remove the K-D flask and allow to cool for at least 10 minutes.

9.2.2.5 Quantiatively transfer the extract to a 10-mL culture tube equipped with a PTFE-lined cap with graduation at 5-mL. Adjust the final volume to 5-mL with hexane. Proceed to analyze by GC/ECD if no cleanup procedures are required. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 50-ng/µL.

9.2.3 Sample Extraction by Automated Soxhlet Procedure B – SW-846 Method 3541

9.2.3.1 Automated Soxhlet Extraction Procedure-B involves Tecator Soxtec equipment, which are capable of handling only 10-g sample aliquots. The set-up consists of three 6-position extractors and two 2-position extractors to handle a total of 22 samples, which will include up to 5 QC samples (MB, LCS, LCSD, MS, and MSD). The five extractors are operated independent using three hot oil baths equipped with independent controllers. The condensers are cooled using a circulating refrigerant bath. Follow manufacturer instructions for setting up and operating the Soxtec equipment.

9.2.3.2 Weight out 10-g of the soil sample to three significant numbers (±0.1-g) into 500-mL beakers. Record the exact weight in the organic Extraction logbook (See Appendix 11 for documentation). Add enough sodium sulfate (10-g) to dry the soil and mix well with a spoon. When the soil is dry (freely flowing powder), transfer the entire contents of the beaker quantitatively to the rigid paper thimbles suspended from the stainless steel holders. Add 1.0-mL of surrogate solution to all the samples (including QC) and 0.25-mL of the appropriate spiking solutions to the LCS/LCSD & MS/MSD samples.

9.2.3.3 Clean and completely assemble the Soxtec extractors. Check the heating oil level in the automated Soxhlet unit and add oil if needed. See service manual for details. Set the temperature on the service unit at 80°C. Press the “MAINS” button and verify that the switch lamp is now in the “ON” position. Set the flow rate for the circulator at approximately 2-L/min to minimize solvent loss from the condensing unit.

9.2.3.4 Immediately transfer the thimbles containing the weighed samples into the condensers. Raise the knob to the "BOILING" position. The magnet will now fasten to the thimble. Lower the knob to the "RINSING" position. The thimble will now hang just below the condenser valve.

9.2.3.5 Insert the extraction cups containing boiling chips, and load each with 50-mL of extraction solvent. Using the cup holder, lower the locking handle, ensuring that the safety catch engages. The cups are now clamped into position. (The seals must be pre-rinsed or pre-extracted with extraction solvent prior to initial use).

9.2.3.6 Move the extraction knobs to the "BOILING" position. The thimbles are now immersed in solvent. Set the timer for 60 minutes. The condenser valves must be in the "OPEN" position. Extract for the preset time.

9.2.3.7 After rinse time has elapsed, close the condenser valves by turning each a quarter-turn, clockwise. When all but 2- to 5-mL of solvent have been collected, open the system and remove the cups.

9.2.3.8 Transfer the contents of the cups to 15-mL graduated, conical-bottom glass tubes attached to the K-D flasks. Rinse the cups using methylene chloride and add the rinsates to the glass tubes.

9.2.3.9 Add two boiling chips to the K-D flask and attach a three-ball Snyder column. Pre-wet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1-ml, add 5-mL of hexane and continue with solvent removal. When the extract reaches 1 to 2-mL remove the K-D flask and allow to cool for at least 10 minutes.

9.2.3.10 Quantiatively transfer the extract to a 10-mL culture tube equipped with a PTFE-lined cap with graduation at 5-mL. Adjust the final volume to 5-mL with hexane. Proceed to analyze by GC/ECD if no cleanup procedures are required. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 50-ng/µL.

9.2.4 Sample Preparation for waste samples

9.2.4.1 If the waste is soluble in extraction solution, add approximately 1.0-g to a 15-mL culture tube. Record the exact weight in the Organic Extraction Log. Add 1.0-mL of the surrogate solution for samples and 1.0mL of surrogate plus 1.0-mL of appropriate spiking solution for LCS/LCSD & MS/MSD samples. Dilute the extract to 10.0 mLs with extraction solution and proceed with GC/ECD analysis. Add 10-uL of the internal standard solution to 1.0-mL of the final extract used for instrumental analysis so that the internal standard concentration is 500-ng/µL.

9.2.4.2 If the waste is not soluble in the extraction solution, the sample must be extracted like a sediment/soil sample using only 1.0-g instead of 30-g. Proceed to cleanup steps if the sample appears to be dirty.

9.2.5 Spiking Solutions - When the presence of specific Aroclors is not anticipated, the Aroclor 1016/1260 mixture is used for spiking LCS, LCSD, MS and MSD samples. When specific Aroclors have been identified in site samples, calibration solutions and spiking solutions based

on the specifc Aroclor may be used for the analysis. Procedues described above should be used with appropriate modification in such instances.

9.3 Extract cleanup - The cleanup options that are available for PCBs analysis have been described earlier. A summary of the sulfuric acid cleanup procedure (See AML SOP based on SW-846 Method 3665A) is described in this SOP.

9.3.1 Transfer 2.0-mL of the sample extract using a gas-tight syringe to a 10-mL culture tube equipped with a Teflon-lined screw cap. Add 5.0-mL of concentrated sulfuric acid, cap it, shake, and centrifuge.

9.3.2 If color persits, transfer 1.5-mL of the sample extract after acid cleanup to another 10-mL culture tube and treat it with 5-mL of potassium permanganate solution.

9.3.3 Transfer 1.0-mL of the upper hexane layer using a gas-tight syringe to a clean test tube. Fortify the extract with internal standard and analyze as described elsewhere.

9.4 GC conditions - This SOP based on the SW-846 Method 8082 and 8082A, which allows the analyst to choose between a single-column or a dual-column configuration in the injector port. Either wide- or narrow-bore columns may be used.

9.4.1 Single-column analysis - This capillary GC/ECD method allows the analyst the option of using 0.25-0.32 mm ID capillary columns (narrow-bore) or 0.53 mm ID capillary columns (wide-bore). The use of narrow-bore (0.25-0.32 mm ID) columns is recommended when the analyst requires greater chromatographic resolution. Use of narrow-bore columns is suitable for relatively clean samples or for extracts that have been prepared with one or more of the clean-up options referenced in the method. Wide-bore columns (0.53 mm ID) are suitable for more complex environmental and waste matrices. This option is not used by AML.

9.4.2 Dual-column analysis - The dual-column/dual-detector approach involves the use of two 30-m fused-silica open-tubular columns of different polarities, thus different selectivities towards the target compounds. The columns are connected to an injection tee and ECD detectors. Dual column is employed at AML.

9.4.3 GC temperature programs and flow rates:

9.4.3.1 A copy of the GC operating conditions for the analysis of PCBs as Aroclors is included in Appendix 1. Use the conditions in this method as guidance and establish the GC temperature program and flow rate necessary to separate the analytes of interest. When determining PCBs as Aroclors, chromatographic conditions should be adjusted to give adequate separation of the characteristic peaks in each Aroclor.

9.4.3.2 The retention times for the various peaks are included in the initial calibration (ICAL) summary, which is included in Appendix 3. Once established, the same operating conditions must be used for the analysis of samples and standards.

9.5 Calibration

9.5.1 Prepare calibration standards as described in the Standards section. Refer to AML SOPs based on EPA SW-846 Method 8000 for proper calibration techniques for both initial calibration and calibration verification. The SW-846 methods should be used when a suitable AML SOP is unavailable during method development and/or implementation. The calibration standards must contain the internal standard at the same concentration as the sample extracts. NOTE: Because of the sensitivity of the electron capture detector, the injection port and column should always be cleaned prior to performing the initial calibration.

9.5.2 When PCBs are to be quantitatively determined as Aroclors, the initial calibration consists of two parts, described below:

9.5.2.1 The standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. Thus, such a standard may be used to demonstrate the linearity of the detector and that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample. Therefore, an initial five-point calibration is performed using the mixture of Aroclors 1016 and 1260.

9.5.2.2 Standards of the other five Aroclors are necessary for pattern recognition. These standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1016/1260 mixture has been used to describe the detector response. The mid-level standards for these five Aroclors should be analyzed before the analysis of any samples. In practice, most samples are free of these Aroclors and the most commonly identified PCB is Aroclor-1260, which is present in the initial calibration mixture. Sample extracts may be analyzed quantitatively for Aroclor-1016 and 1260 and screened for the presence/absence of other Aroclors.

9.5.2.3 If any Aroclor other than 1016 and 1260 appears to be present, the routine single-point calibration performed to establish response factors and chromatography pattern may also be used to perform preliminary quantitation for these five Aroclors. For DoD projects, reanalysis of the sample extracts using a special five-point initial calibration for the identified Aroclor is required. Preliminary quantitation based on the single mid-point calibration may be used to determine the appropriate dilution that may be necessary for reanalysis.

9.5.2.4 In situations where only a few Aroclors are of interest for a specific project, the analyst may employ a five-point initial calibration of each of the Aroclors of interest (e.g., five standards of Aroclor 1232 if this Aroclor is of concern) and not use the 1016/1260 mixture described in this SOP. The spiked sample

analyses may also be performed (see Section 5.9.3) using the Aroclor of interest instead of the default Aroclors.

9.5.3 Establish the GC operating conditions as described above. Optimize the instrumental conditions for resolution of the target compounds and sensitivity. A 2-uL injection of each calibration standard is recommended. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest. Once established, the same opearting conditions must be used for the analysis of samples and standards.

9.5.4 Record the peak area (or height) for each congener (not used at AML) or each characteristic Aroclor peak to be used for quantitation.

9.5.4.1 Choosing five peaks (minimum required is 3 per SW-846 Method 8082 and 8082A) was recommended in an earlier version of the AML SOP. However, using small peaks that make up <25% of the largest peak caused higher variabilities for the quantitation. Choosing only three peaks for each Aroclor is recommended unless five peaks are required to satisfy specific clients.

9.5.4.2 The chosen peaks must be characteristic of the Aroclor in question. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of chosen peaks should include at least one peak that is unique to that Aroclor. Use a total of six to ten peaks (three to five peaks for each Aroclor, 1016 and 1260), none of which should be found in both of these Aroclors.

9.5.4.3 This discussion applies both to the primary column and the confirmation column. See Appendix 3 for a summary of the initial calibration and the list of peaks used for Aroclor 1016 and 1260.

9.5.4.4 Late-eluting Aroclor peaks are generally the most stable in the environment.

9.5.5 When determining Aroclors by the internal standard procedure (used at AML), calculate the response factor (RF) for each peak in the calibration standards relative to the internal standard using the equation that follows.

$$RF = \frac{A_s \text{ x } C_{is}}{A_{is} \text{ x } C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.

A_{is} = Peak area (or height) of the internal standard.

C_s = Concentration of the analyte or surrogate, in µg/L.

C_{is} = Concentration of the internal standard, in µg/L.

9.5.6 When determining PCBs as Aroclors by the external standard technique (seldom used at AML), calculate the calibration factor (CF) for each

characteristic Aroclor peak in each of the initial calibration standards using the equation below.

$$CF = \frac{\text{Peak Area (or Height) in the Standard}}{\text{Total Mass of the Standard Injected (in nanograms)}}$$

Three to five sets of calibration factors will be generated for the Aroclor-1016 and Aroclor-1260, each set consisting of the calibration factors for each of the peaks chosen for this mixture. The single standard for each of the other Aroclors will generate calibration factors for each of the selected peaks.

9.5.7 The response factors or calibration factors from the initial calibration are used to evaluate the linearity of the initial calibration. Calculate the mean response or calibration factor, the standard deviation, and the relative standard deviation (RSD) for each Aroclor peak.

9.5.7.1 The 8082 and 8082A methods do not provide detailed guidance for handling the initial calibration data other than making the reference to SW-846 Method 8000. These methods and the chromatographic data systems are intended for the analysis of single-component analytes. The following guidance is used at AML. The analyst should obtain additional guidance from the QA Director or the Operations Manger to handle specific requests from clients.

9.5.7.2 Ideally, the %RSD for each Aroclor peak should be less than 20% to assume linearity. If five peaks are chosen and the %RSD is greater than 20% for any of the five Aroclor peaks, the mean %RSD for all five peaks should be less than 20%. This criteria applies to each Aroclor (1016 and 1260) present in the ICAL as well as any other Aroclor that may be run at five different concentrations.

9.5.7.3 In this SOP, each peak is treated as a separte analyte, although they are not used alone to report sample quantities for the Aroclor. If linearity cannot be demonstrated for all five peaks, one of the following options may be used:

9.5.7.3.1 Use three or four strong, better-performing peaks rather than using all five peaks for quantitation.

9.5.7.3.2 Use least square regression methods to calculate sample concentrations. For regression methods, the instrument response is treated as the dependent variable (y) and the standard amount is the independent variable (x). When “Cs” is used as “x”, use “As*Cis/Ais” as “y”. When

"Cs/Cis" is used as "x", use "As/Ais" as "y". Do not include origin (0,0) as an extra calibration point.

9.5.7.3.3 Weighted least squares method is preferred for calculating concentrations more accurately at lower levels. A linear least squares equation (y = a*x + b) is preferred over non-linear calibration equations (y = a*x2 + b*x + c). The coefficient of determination (COD), coefficient of variation (r2) or correlation coefficient (r) should be used to measure the goodness of fit. For regression models to be used for quantitative purposes, the COD, r2 or r must be greater than or equal to 0.99. A value of 1.00 indicates a perfect fit.

9.5.7.3.4 Corrective action may be required if the criteria for %RSD, r, r2, or COD are not met. In practice, it may be better to check and correct the instrument and/or calibration standards for potential problems and reanalyze the ICAL standards to meet linearity criteria.

9.5.7.3.5 An initial calibration (ICAL) should be considered a single event process. Consult the QA office or check applicable EPA methods (e.g. SW-846 Method 8000C and 8081A) for additional guidance on appropriate corrective action.

9.5.7.3.6 Re-fitting of ICAL – The selected model for ICAL may be subjected to an additional re-fitting check to establish representativeness of the model for each compound in each ICAL standard. This is an optional procedure, which is recommended but not required since automated data processing procedures may not be able to accommodate it. Calculate the %D between the calculated amount (Cc) for each compound in each ICAL standard from the ICAL model and its expected value (Ce). Ideally, the %D ((Cc-Ce) * 100 /Ce) for each compound in ICAL standard should meet the QC criteria for %RSD. It is possible for one or more ICAL standards to have %D exceeding the %RSD criteria, while the %RSD for ICAL model is within QC limits. The re-fitting procedure may be used to identify ICAL standards that need re-analysis or dropped from the calculation of ICAL model when re-analysis was not performed.

9.5.7.3.7 Check and optimize instrument operating conditions including automated peak integration procedures for appropriateness in addressing the chromatographic needs of the specific compound with problems.

9.5.7.3.8 If the problem appears to be associated with just one of the ICAL standards, that one standard may be reanalyzed once immediately (must be within 8-hours) after the ICAL. Discard all the results from the original analysis of the standard in question and replace it with the results from re-analysis.

9.5.7.3.9 When additional calibration standards (six or seven instead of five) are analyzed, narrowing the range of concentrations to be used for determining the goodness of fit is an acceptable procedure. Five or more standards should remain for the calculation of RSD or linear regression for each compound after one or more points are removed from the ICAL. More points (6 for second order and 7 for third order) may be needed for non-linear regression. It is generally prohibited to remove data points from within a calibration range, while still retaining the extreme ends of the calibration range. Do not discard a middle point unless it appears to have an obvious problem and six or more standards were analyzed for ICAL and re-analysis of the affected standard was not performed within the required time limits. Changes to the upper end of the calibration range will affect the need to dilute samples above the range. A change to the lower range is not recommended since it will affect the method quantitation limit (MQL) used to report data. The default MQLs, which are based on the lowest ICAL concentration are stored in the LIMS and project-specific changes are not practical.

9.5.7.3.10 The refitting procedures may lead to failed %Ds for upper level calibration standards for average response factor methods. The analyst may take alternate corrective actions such as the analysis of sample extracts under dilution for the affected compounds.

9.5.7.3.11 The refitting procedures may lead to failed %Ds for lower level calibration standards for regression methods. The analyst may take appropriate corrective action such as flagging the sample result as an estimated amount ("J" flag) at the lower level encountered in the sample.

9.5.7.3.12 The term "calibration verification" (CALV) is used in this SOP to describe initial verification (ICV) of the initial calibration (ICAL) using second-source standards and subsequent continuing verifications (CCVs) using same-source standards. The response

factors calculated during CALV are not used for sample quantitation since CALV standards are not used as "continuing calibration" standards permitted in some of the EPA methods.

9.5.7.3.13 Initial Calibration Verification (ICV) - Analysis of a second source ICV standard is required before any samples can be analyzed for DoD projects. Since second source standards are not employed for the analysis of LCS/LCSD and MS/MSD samples, ICV analysis is applicable for all projects. Calculate the percent difference (%D) or %Drift for the observed response factor in ICV from the mean RF as described for CCV. The %D or %drift should not exceed ±15% for each target compound. Document calibration verification in instrument printouts as described for the CCV. Check the ICAL and ICV standards if it is not achievable for all the target compounds without any exception. The ICV may be used as the beginning CCV for sample analysis.

9.5.7.3.14 If the above criteria are met, the analysis for analytical samples can begin.

9.6 Retention time windows

9.6.1 Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for the identification of PCBs as Aroclors. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability.

9.6.2 The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that cannot be confirmed upon further analysis.

9.6.3 Establish retention time windows based on multiple injection of the calibration standards over several days. Establish mean, standard deviation (SD), ± 2*SD for warning limits, and ±3*SD control limits for retention time window for each peak. See Appnedix 2 for more details about this procedure. Use professional judgment to widen the window beyond the control limits, if necessary.

9.7 Gas chromatographic analysis of sample extracts

9.7.1 The same GC operating conditions used for the initial calibration must be employed for samples analyses. Verify calibration each 12-hour shift by injecting calibration verification standards prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than once every 12-hours and at the end of the analysis sequence. Client specification and good laboratory practice at AML require the analysis of CCVs after every 10 samples (after every 5 samples if dirty extracts are involved) to minimize the number of samples requiring re-injection when QC limits are exceeded. A beginning CCV is also required if sample analyses are not performed immediately after an ICV following an ICAL. An ending CCV is always required.

9.7.2 For Aroclor analyses, the calibration verification standard should be a mid-level mixture of Aroclor 1016 and Aroclor 1260. The calibration verification process does not *require* analysis of the other Aroclor standards used for pattern recognition, but the analyst may wish to include a standard for one of these Aroclors after the 1016/1260 mixture used for calibration verification throughout the analytical sequence.

9.7.2.1 The response factor, RF_v (or calibration factor, CFv) for each analyte calculated from the continuing calibration verification standard must not exceed a difference of more than ±15% when compared to the mean response factor, RF_m (or calibration factor, CF) from the initial calibration curve.

$$\% \text{ Difference} = \frac{(RF_m - RF_v) * 100}{RF_m}$$

$$\% \text{ Difference} = \frac{\overline{CF} - CF_v}{\overline{CF}} \times 100$$

9.7.2.2 If this criterion is exceeded for any calibration factor or response factor, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis. Document calibration verification on instrument printouts as described in Appendix 4.

9.7.2.3 If routine maintenance does not return the instrument performance to meet the QC requirements based on the last initial calibration, then a new initial calibration should be performed.

9.7.3 Inject a 2-uL aliquot of the concentrated sample extract.

9.7.4 Qualitative identifications of target analytes are made by examination of the sample chromatograms, as described in the following sections.

9.7.5 Quantitative results are determined for each identified analyte (Aroclor), using the procedures described in the following sections for either the internal or the external calibration procedure. If the responses in the sample chromatogram exceed the calibration range of the system, dilute the extract and reanalyze. Peak height measurements are recommended over peak area when overlapping peaks cause errors in area integration.

9.7.6 Each sample analysis must be bracketed with an acceptable initial calibration, calibration verification standard(s) (each 12-hour shift), or calibration standards interspersed within the samples. When a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that last met the QC criteria must be re-injected.

9.7.7 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements. It is *recommended (and used at AML)* that standards be analyzed after every 10 samples (*required* after every 20 samples and at the end of a set) to minimize the number of samples that must be re-injected when the standards fail the QC limits. The sequence ends when the set of samples has been injected or when qualitative or quantitative QC criteria are exceeded.

9.7.8 Use the calibration standards analyzed during the sequence to evaluate retention time stability. If any of the standards fall outside their daily retention time windows, the system is out of control. Determine the cause of the problem and correct it.

9.7.9 If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract or replacement of the capillary column or detector is warranted. Rerun the sample on another instrument to determine if the problem results from analytical hardware or the sample matrix.

9.7 Qualitative identification

The identification of PCBs as either Aroclors or congeners using this method with an electron capture detector is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes. Tentative identification of an analyte occurs when a peak from a sample extract falls within the established retention time window for a specific target analyte. Each tentative identification must be confirmed using a second GC column of dissimilar stationary phase (as in the dual-column analysis), based on a clearly identifiable Aroclor pattern. The Aroclor pattern for the seven Aroclors described in this SOP are included in Appendix 4.

9.7.1 When simultaneous analyses are performed from a single injection, it is not practical to designate one column as the analytical (primary) column and the other as the confirmation column. Since the calibration standards are

analyzed on both columns, the results for both columns must meet the calibration acceptance criteria. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed. Perform quantitation on both columns as described in Section 9.8.

9.7.2 **Special Analysis** - When samples are analyzed from a source known to contain specific Aroclors, the results from a single-column analysis may be confirmed on the basis of a clearly recognizable Aroclor pattern ***when specifically requested by the Client to support rapid turnaround and/or high sample throughput requirements.*** NOTE: ***This approach should not be used for routine evaluation of site samples for PCBs. This approach should not be attempted for samples from unknown or unfamiliar sources or for samples that appear to contain mixtures of Aroclors.*** In order to employ this approach, the analyst must document the following: (1) The peaks that were evaluated when comparing the sample chromatogram and the Aroclor standard. (2) The absence of major peaks representing any other Aroclor. (3) The source-specific information indicating that Aroclors are anticipated in the sample (e.g., historical data, generator knowledge, etc.). (4) This information should either be provided to the data user or maintained by the laboratory.

9.7.3 GC/MS confirmation may be performed if the concentration of the Aroclors are atleast 100 times the lowest point on the calibration curve for SVOCs (See AML SOP based on EPA SW-846 Method 8270C).

9.8 Quantitation of PCBs as Aroclors

The quantitation of PCB residues as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample as described in the section dealing with qualitative identification (Section 9.7).

9.8.1 Use the individual Aroclor standards (not the 1016/1260 mixtures) to determine the pattern of peaks on Aroclors 1221, 1232, 1242, 1248, and 1254. The patterns for Aroclors 1016 and 1260 will be evident in the mixed calibration standards. See Appendix 4 for a library of the Aroclor patterns.

9.8.2 Once the Aroclor pattern has been identified, compare the responses of 5 major peaks in the calibration standard for that Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the mean individual response factor (calibration factor for external standard) for each of the 5 characteristic peaks chosen for Aroclor 1016 and 1260. If five point calibrations are run using any of the other Aroclors, use the mean RF for each peak. A concentration is determined using each of the characteristic peaks and then those 5 concentrations are averaged to determine the concentration of that Aroclor.

9.8.3 If multi-point calibrations are not routinely performed (e.g. Aroclors 1221, 1232, 1242, 1248, and 1254), determine the preliminary concentration of these Aroclors using the RFs established based on the single point. Perform

this procedure for each Aroclor peak. A preliminary concentration is determined using each of the characteristic peaks and then those 5 concentrations are averaged to determine the mean preliminary concentration of that Aroclor.

9.8.4 For DoD projects, quantitation of Aroclors 1221, 1232, 1242, 1248, and 1254 when present should be performed using a multi-point ICAL similar to that used for Aroclor 1016/1260 for routine analysis. The sample extracts should be reanalyzed using a multi-point ICAL for these Aroclors on the same instrument that was used originally to analyze the associated QC samples (MB, LCS/LCSD, and MS/MSD). The preliminary concentration established from routine analysis may be used to determine the dilution necessary for re-analysis. Reanalysis of the associated QC samples is not required unless it is performed on a different instrument.

9.8.5 Repeat the procedure to calculate Aroclor concentrations for both chromatographic columns. Calculate the percent RPD of the two concentrations and compare it to the EPA SW-846 Method 8000 criteria. For all projects including the DoD projects, report the higher of the two values for the analyte under consideration.

9.8.5.1 If the RPD is less than 40%, report the higher of the two values without qualifications. Even if visible interference is present for one or more peaks in one of the columns, disregard this information.

9.8.5.2 If the %RPD is greater than 40%, report the higher of the two values as an estimated value ("J" flag). If visible interference is present for the column reported, document this problem on the case narrative and include the value from the other column in the case narrative, when it is required.

9.8.5.3 **Special Situations** – Client driven procedures may be used for reporting data from dual column analyses for special projects (e.g. composite input water samples from groundwater pump and treat systems). For these projects, reporting the higher of the two values may introduce unwanted fluctuations in analyte sample data over time, which may not be supported by groundwater analytical data gathered for other analytes from the same samples.

9.8.5.3.1 For these projects, an interferece-free column may be designated as the primary column and data may be reported from this column.

9.8.5.3.2 When strong and visible interference for one or more Aroclor peak is noticed that results in a positive bias for the column reported, data from the other column (confirmation) may be reported for the affected analyte.

9.8.5.3.3 Use professional judgment for selecting value to report, if interference is suspected and document it in the case narrative.

9.8.5.3.4 Report data without qualifications if the %RPD is <40% and as an estimated value (“J” flag) if the RPD is >40%.

9.8.6 Weathering of PCBs in the environment and changes resulting from waste treatment processes may alter the PCBs to the point that the pattern of a specific Aroclor is no longer recognizable. Samples containing more than one Aroclor present similar problems. Use professional judgment in the reporting of data for these samples. Clients may be consulated for handling this situation.

9.9 GC/MS confirmation may be used in conjunction with either single-or dual-column analysis if the concentration is sufficient for detection by GC/MS. Full-scan quadrupole GC/MS will normally require a higher concentration of the analyte of interest than selected ion monitoring techniques. The concentrations will be instrument-dependent, but values for full-scan quadrupole GC/MS may be as high as 500-ng/µL in the final extract since Aroclors consist of a number of compounds, whose total add up to the concentration described. The GC/MS must be calibrated for the specific target analytes. When using SIM techniques, the ions and retention times should be characteristic of the Aroclors to be confirmed. GC/MS confirmation should be accomplished by analyzing the same extract used for GC/ECD analysis and the extract of the associated blank. The base/neutral/acid extract (if also run on the same sample) and the associated blank may be used for GC/MS confirmation if the surrogates and internal standards do not interfere. However, if the compounds are *not* detected in the base/neutral/acid extract, then GC/MS analysis of the pesticide extract should be performed.

9.10 Chromatographic System Maintenance as Corrective Action - When system performance does not meet the established QC requirements, corrective action is required, and may include one or more of the following.

9.10.1 Splitter connections- For dual columns which are connected using a press-fit Y-shaped glass splitter or a Y-shaped fused-silica connector, clean and deactivate the splitter port insert or replace with a cleaned and deactivated splitter. Break off the first few inches (up to one foot) of the injection port side of the column. Remove the columns and solvent backflush according to the manufacturer's instructions. If these procedures fail to eliminate the degradation problem, it may be necessary to deactivate the metal injector body and/or replace the columns.

9.10.2 Metal injector body - Turn off the oven and remove the analytical columns when the oven has cooled. Remove the glass injection port insert (instruments with on-column injection). Lower the injection port temperature to room temperature. Inspect the injection port and remove any noticeable foreign material.

9.10.2.1 Place a beaker beneath the injector port inside the oven. Using a wash bottle, rinse the entire inside of the injector port with acetone and then rinse it with toluene, catching the rinsate in the beaker.

9.10.2.2 Consult the manufacturer's instructions regarding deactivating the injector port body. Glass injection port liners may require deactivation with a silanizing solution containing dimethyldichlorosilane.

9.10.3 Column rinsing - The column should be rinsed with several column volumes of an appropriate solvent. Both polar and nonpolar solvents are recommended. Depending on the nature of the sample residues expected, the first rinse might be water, followed by methanol and acetone. Methylene chloride is a good final rinse and in some cases may be the only solvent required. The column should then be filled with methylene chloride and allowed to stand flooded overnight to allow materials within the stationary phase to migrate into the solvent. The column is then flushed with fresh methylene chloride, drained, and dried at room temperature with a stream of ultrapure nitrogen.

10.0 QUALITY CONTROL

10.1 Quality control procedures are used to ensure that proper sample preparation techniques, extract cleanup procedures, and instrument calibration procedures are performed. This involves the analysis of method blanks (MB), laboratory control samples (LCS), and matrix spikes (MS).

10.2 The frequency of analysis of LCS analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples. LCS can be run in duplicate to obtain precision (RPD) information. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. Document LCS results in Form-1 equivalents and recoveries in Form-3 Equivalents (See Appendix 8 for more details).

10.3 The frequency of analysis of MS analysis is equivalent to a minimum of 2 per 20 samples or 2 per batch if less than 20 samples. MS is run in duplicate to obtain precision (RPD) information. Document MS results in Form-1 equivalents and recoveries/RPDs in Form-3 Equivalents (See Appendix 7 for more details).

10.4 The frequency of analysis of MB analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples.

10.5 Include a calibration standard after each group of 20 samples (it is *recommended* that a calibration standard be included after every 10 samples to minimize the number of repeat injections) in the analysis sequence as a calibration check. The response factors for the calibration should be within 15 percent of the initial calibration. When this continuing calibration is out of this acceptance window, the laboratory should stop analyses and take corrective action.

10.6 Whenever quantitation is accomplished using an internal standard, internal standards must be evaluated for acceptance. The measured area of the internal standard must be no more than 50 percent different (-50% to +100%) from the

area obtained during initial calibration. When the internal standard peak area is outside the limit, all samples that fall outside the QC criteria must be reanalyzed.

10.7 Sample Quality Control for Preparation and Analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). This includes the addition of surrogates to each field sample and QC sample and evaluating their recoveries against QC limits. The surrogate recoveries are documented using Form-2 equivalents (See Appendix 9 for more details).

10.8 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair, spiked with the Aroclor 1016/1260 mixture. However, when specific Aroclors are known to be present or expected in samples, the specific Aroclors should be used for spiking. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. Since this knowledge may not always be available, AML will always analyze the MS in duplicate (MS and MSD). Sample duplicate analyses are not performed.

10.9 Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

11.0 **DATA VERIFICATION AND VALIDATION -** This section will provide guidance and procedures to assess if data produced using this SOP is valid and legally defensible. Document review and comments on the checklists (See Appendix 5). Verify that sample preparation logs are complete and the instrument run logs are available for review (See Appendix 11). Anomalies and exceptions should be documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on the checklists. Army (USACE) and Air Force (AFCEE) projects may have special requirements (See Appendix 13) that will require extra attention. A simple, universal procedure for evaluating and choosing appropriate corrective action doe not exist to deal with situations when one or more QC parameters are outside the acceptance window. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints. To make the matters simple, AML has adopted the QC limits provided in the DoD QSM as default limits for the surrogate, LCS, and MS recoveries.

11.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) projects may have additional requirements that are related to USACE laboratory validation (CX), USACE District requirements, government contract requirements, specific USACE

project requirements or USACE Architect/Engineer (A/E) requirements. USACE-CX requirements (Summary of Method Quality Objectives for Method 8082) are included in Appendix 13.

11.1.1 The USACE requirements shown in Appendix 13 and elsewhere in this SOP are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may or may not be attainable for routine sample analysis. Since USACE MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

11.1.2 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality objectives and/or other QC requirements. Exceptions and data qualifications necessary should be documented on the checklists and eventually on case narratives.

11.1.3 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist, perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete. For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete. For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director will review the checklists (initial/date as necessary), prepare case narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

11.1.4 The blank criteria shown in Appendix 13 has been modified by USACE auditor(s) as one half of the applicable PQL (personal communications, USACE auditor, 1999).

11.2 HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

11.2.1 WATER - The holding times for PCBs is 7 days from sampling.

11.2.2 SOIL – A 14 day holding is used for soil and waste samples.

11.2.3 Holding times are established by comparing the chain of custody sampling date with the date of the injection after purging.

11.2.4 ACTION – If holding times have been exceeded, contact the client. All positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or innvalid (I).

11.3 INITIAL CALIBRATION – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the GC/ECD is capable of producing data within a range that is linear. Verification of the ICAL with a second source standard (ICV) is required for USACE and AFCEE projects (See Appendix 13). The ICV is performed and assessed similar to CCV (Section 11.4) except for special USACE and AFCEE requirements.

11.3.1 Compounds – Check to see if any Aroclor peak is outside the criteria of 20% or less for %RSD. If an Aroclor peak is outside the 20% criteria, check to see whether the mean %RSD for the three or five Aroclor peaks is within the 20% criteria.

11.3.2 ACTION - Use regression procedures for peaks not meeting the criteria, if the mean %RSD is within criteria. The "r" , “r_2” or “COD”, the measure of the fit for regression procedures should be >0.99. Apply criteria to each Aroclor and to data from both columns.

11.3.3 ACTION – Check and correct the standards, including the range of concentrations for appropriateness and repeat the ICAL analysis.

11.3.4 ACTION – Use a lesser number of peaks (minimum 3) if the criteria are not met for five peaks (desirable) selected for each Aroclor.

11.3.5 ACTION - Report the problem to the Project Manager and report detects and non-detects with aapropriate qualifiers for rapid TAT projects. Mention the ICAL anomaly in the case narrative.

11.4 INITIAL CALIBRATION VERIFICATION – Calibration verification includes initial calibration verification (ICV) and continuing calibration verifications (CCVs). A second source ICV is used to check the integrity of the ICAL standards used for calibrating the instrument. The 12-hour analytical shift begins with the injection of the ICV (instead of a CCV) for sample analyses that are performed immediately after an ICAL.

11.4.1 Target Compounds - Check to see if %D for any of the target compounds is outside the USACE QC criteria (<±15%)

11.4.2 ACTION - Check the ICAL and ICV standards for integrity. Replace one or more of the working standards (ICV first and ICAL later) for trouble

shooting. Correct the problem(s) and repeat ICV analysis. Use professional judgment for non-USACE projects.

11.4.3 ACTION - When ICAL and ICV are acceptable based on SW-846 criteria, positive results and non-detects for target compounds not meeting the ±15% %D may be reported without data qualifications for non-USACE projects provided a valid CCV was obtained immediately following the CCV. The ICAL and/or ICV standards should be inspected and replaced, if necessary for compliance with this requirement for USACE projects.

11.5 CONTINUING CALIBRATION VERIFICATION – The continuing calibration verification (CCV) is used to check that the instrument is still operating under the linear range of the initial calibration and has remained stable every 12 hours.

11.5.1 IS area counts (use mean IS area from ICAL for the %D of IS area) must not vary by more than a factor of 2 (-50% or + 100%) from the associated ICAL.

11.5.2 Compounds – Check to see if the %D for any Aroclor peak is outside the criteria of 15% or less. Check to see that the mean %D for all the peaks is <15%.

11.5.3 The %D for Aroclors present in the sample – The %D for the CCVs for each Aroclor peak before and after the sample analysis should be within QC limits when internal standards are not used. The %D for the CCV before sample analysis should be within QC limits when internal stanadrds are used.

11.5.4 The %D for Aroclors not present in the sample – The %D for the CCVs before and after the sample analysis may be >(+15%). Corrective action is not required unless the %D is < (-15%).

11.5.5 ACTION – Correct the problem and rerun the calibration verification and all associated samples. Positive results for compounds not meeting the criteria may be qualified as estimated (J) by the user (client). Report non-detects without qualifiers and mention these outliners in the case narrative.

11.6 METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Sample data qualification to alert blank contamination is performed by the laboratory. Sample data qualification based on all the blank results (laboratory MB, equipment blank, and other field blanks) is performed by the user (client). The processing of blanks helps eliminate reporting of false positives in project reports.

11.6.1 Method blanks should be free of all target anaytes and other interfering non-target analytes. To be acceptable, blank results should not exceed one half of the applicable MQL for any compound.

11.6.2 Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to

analyze associated samples. If levels greater than one half the PQLs are present, inspect and correct the problem. Reanalyze all associated samples. Contact client if other criteria are applicable. The only exception may be quick turn around projects.

11.6.3 ACTION – All positive results are reported with a "B" code, if contaminants are found in the method blank.

11.6.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, then no action is taken.

11.6.5 ACTION – Any compound detected in the sample which were also detected in associated method blank at levels greater than one half the PQLs should be re-extracted unless other client criteria are applicable.

11.6.6 ACTION – If contaminants resulting in GC/ECD peak saturation are present, all affected compounds are qualified as invalid (I).

11.7 SURROGATE RECOVERIES – Surrogates or system monitoring compounds are different structurally, but similar chemically to classes of target compounds. These surrogate compounds are added just prior to extraction of the samples and the percent recoveries of the surrogates are calculated and evaluated. The SW-846 method and DoD QSM recommeds the use of Decachlorobiphenyl as the only surrogate for PCBs analysis. The AML procedure involves sharing of a sample extract for the analysis of organochlorine pesticides (8081) and PCBs (8082), which are method analytes for other EPA methods (508.1 and 608). Both DCB and tetrachloro-m-xylene (TCMX) are used as surrogates and the QC limits recommended for DCB and TCMX for Method 8081 are also extended to the PCBs analysis, although sample extracts undergo different cleanup methods for the two procedures.

11.7.1 Document surrogate recoveries in Form-2 equivalents (See Appendix 9 for detials). Verify that the surrogate recoveries for both surrogate compounds (DCB and TCMX) are within QC limits (DoD QSM) for both surrogates) Flag outliers with an asterisk (*).

11.7.2 ACTION – When surrogate recoveries are outside control limits the sample must be re-analyzed. Reanalysis may be performed under dilution if original analysis should indicate high levels of target and non-target analytes. Reextraction and cleanup will be necessary if reanalysis does not resolve the problem. If surrogate recoveries are still outside the limits, report both sets of data and described the problem in the case narrative.

11.8 LABORATORY CONTROL STANDARD (LCS) – A LCS analysis is used to evaluate method competence in the absence of matrix effects. The LCS should be from a second source standard. The LCS may provide a mechanism to check method efficiency for target compounds without the sample matrix as a factor. See project plan and other available information (e.g. Appendix 13) for specific client requirements.

11.8.1 The QC limits for LCS recoveries are shown in Tables 1 and 2.

11.8.2 Check the results for the LCS recoveries to see if the results are within the acceptable range. Flag outliers with an asterisk (*).

11.8.3 ACTION – If deviations are noticed, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements, which may be compound specific. Check with the project manager about such requirements. Professional judgment may be used by the user in evaluating sample data qualification options in conjunction with other QC data for the project.

11.9 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike data are used to evaluate the precision and accuracy of the analytical method on project samples. See project plan and other available information (e.g. Appendix 13) for specific client requirements.

11.9.1 The QC limits for MS/MSD percent recoveries are shown in Tables 1 and 2. The QC limits for relative percent difference (RPD) between the MS and MSD results are also shown in these tables.

11.9.2 Check the results for the MS/MSD recoveries. Flag outliers with an asterisk (*).

11.9.3 Perform one manual calculation using raw data and extraction sheet information.

11.9.4 ACTION – No action is taken on MS/MSD recoveries data alone to qualify all associated samples. A general rule is to have at least 90% of the target analytes within the control limits. Since matrix interference could lead to lead to serious analytical problems, the supervisor should be consulted when significant outliers are noted. Document QC outliers for %recovery and RPD on the checklist and on client reports (*) and draw attention to the QC outliers on the case narrative. No action may be taken on MS/MSD recovery data alone either to reanalyze samples and/or to qualify associated samples either by the analyst or by the end user. Professional judgment may be used by the user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

11.10 INTERNAL STANDARD PERFORMANCE – Internal Standard (IS) performance criteria ensures that the GC sensitivity and response is stable during each run.

11.10.1 IS area counts (use mean IS area from ICAL for the %D of IS area) must not vary by more than a factor of 2 (-50% or + 100%) from the associated ICAL/Calibration Verification standard. The internal standard areas for the CCV should not differ by a factor of 2 (-50% or + 100%) from the initial calibration of the same standard.

11.10.2 The retention time of the internal standard must not vary by more than 30 seconds from the associated daily calibration standard.

11.10.3 ACTION – If an internal standard is outside of QC limits of the associated daily standard, then re-analysis is required or a dilution may be required if it appears that interference's occur from target and non-target analytes at high concentrations relative to the ICAL. Do not dilute the sample to the point where all target analytes are non-detect. Professional judgement must be used when dilutions are required for IS criteria.

11.10.4 ACTION – If an internal standard is outside of QC limits of the associated ICAL or CCV standard, correct the problem and re-analyze samples with a valid CCV. If re-analysis has been performed and it is still out, it is of the professional judgement of the analyst to dilute or not to dilute the sample further so as to not to dilute out any target analytes.

11.10.5 ACTION - Report results without qualification describing the QC problem in the case narrative. The user (client) may qualify positive results for compounds quantitated using the affected IS as estimated (J). If a very low IS area is reported, indicating a loss of sensitivity, non-detects may require qualification as invalid (I) or rejected (R). Document recommendation for the client in the case narrative.

11.10.6 ACTION – If an IS retention time varies by more than +/- 30 seconds, the total ion profile must be checked for any false positives or negatives. If a large shift has occurred, then professional judgement must be used as to whether reanalyze or not. See previous section for guidance.

11.11 TARGET COMPOUND IDENTIFICATION – Criteria for GC/ECD qualitative analysis is to minimize the number of erroneous positive or negative identifications of compounds.

11.11.1 Professional judgement must be used when applying the criteria for GC/ECD analysis of target analytes. Take into account factors such as matrix effect and column bleed.

11.11.2 Positive compounds must be within +/- 0.06 relative time (RRT) units of the standard RRT. The results should be >MDL or other threshold for reporting positives (e.g. LLR, RL) on both columns.

11.11.3 The RPD for sample amounts from the two columns for identified compounds should be within <40%. Calculate RPD using Form-10 equivalents (See Appendix 6 for more details).

11.11.4 ACTION - If the RPD for positive results are <40%, report the result from the primary column without any flag. Report results greater than the upper limit of calibration with as estimated with an "E" flag.

11.11.5 ACTION - If the RPD is >40%, report the result from the primary column with a "J" flag. The result from the confirmation column may be reported with a "J" flag at the discretion of the analyst, if intererence is suspected for the compound of interest in the primary column.

11.11.6 ACTION - Samples with E coded data will require reanalysis with dilution. Both sets of results may need to be reported for some clients. See Table 1 and 2 for a summary of the MDLs, LLRs and MQLs.

11.11.7ACTION - Adjust sample results and associated MDL, LLR, and MQL, for percent moisture and non-standard sample weights

11.11.8ACTION - Report sample data in Form-1 equivalents (Appendix 12).

12.0 REFERENCES

12.1Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8081A, Organochlorine Pesticides by Gas Chromatography, December 1996.

12.2Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8082, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, December 1996.

12.3Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8082A, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Draft Revision 1A, May 1999.

12.4Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.

12.5Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

12.6Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3500B, Organic Extraction and Sample Preparation, December 1996.

12.7Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3520C, Continuous Liquid-Liquid Extraction, December 1996.

12.8Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3510C, Separatory Funnel Liquid-Liquid Extraction, December 1996.

12.9Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3511, Organic compounds in water by microextraction, Revision 0, November 2002.

12.10 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3540C, Soxhlet Extraction, December 1996.

12.11 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3541, Automated Soxhlet Extraction, December 1996.

12.12 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3545, Pressurized Fluid Extraction, December 1996.

12.13 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3550B, Ultrasonic Extraction, December 1996.

12.14 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3570, Microscale Solvent Extraction (MSE), Revision 0, November 2002.
12.15 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3600C, Cleanup, December 1996.
12.16 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3640A, Gel-Permeation Cleanup, December 1996.
12.17 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3660B, Sulfur Cleanup, December 1996.
12.18 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3665A, Sulfuric Acid/Permanganate Cleanup, December 1996.
12.19 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3580A, Waste Dilution, December 1996.
12.20 Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, EPA, 40 CFR Part 136, Method 608: Chlorinated Pesticides and PCBs, October 1984.
12.21 Methods for the Determination of Organic Compounds in Drinking Water, Supplement II, EPA/600/4-88-039, Method 508, Determination of Chlorinated Pesticides in Water by Gas Chromatography with an Electron Capture Detector, July 1991.
12.22 Methods for the Determination of Organic Compounds in Drinking Water, Method 508.1, Determination of Chlorinated Pesticides in Water by Gas Chromatography with an Electron Capture Detector.
12.23 USEPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-media, Multi-concentration, OLM02.0.
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12.27 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.
12.28 AML Standard Operating Procedures, Q-Water-RC.
12.29 AML Standard Operating Procedures, Q-WasteMgmt-RD.
12.30 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
12.31 AML Standard Operating Procedures, Q-Controlcharts-RC.

TABLE 1
Aqeous MDLs, LLRs, MQL and QC Limits for LCS/LCSD and MS/MSD Recoveries and RPDs Surrogate Recovery Limits

Polychlorinted Biphenyl (PCBs) Aroclors Analysis by GC/ECD

Analytical Management Laboratories

Preparation Method:	3510C													
Analysis Method:	8082													
	MDL	**MDL**	**MDL**	**MDL**	**LLR**	**MQL**	**Ratio (RQ)**		**DoD QSM LCS & MS Control Limits**		**DoD QSM LCS & MS "ME" Limits**		**RPD**	
Compound	Spike	DB-5	DB-1701	Report	Report	Report	QL/DL	Flag	Lower	Upper	Lower	Upper	Limit	
	ug/L	ug/L	Ug/L	ug/L	ug/L	ug/L			**%R**	**%R**	**%R**	**%R**	%	
Aroclor-1016	1.0	0.13	0.27	**0.27**	0.5	**2.0**	7.5		25	145	25	145	50	
Aroclor-1260	1.0	0.45	0.55	**0.55**	0.5	**2.0**	3.6		30	145	30	145	50	
Other Aroclors:									Default AML Control Limits		Default AML "ME" Limits			
Aroclor – 1221	1.0	0.29	0.41	**0.41**	0.5	**2.0**	4.9		25	145	25	145	50	
Aroclor – 1232	1.0	0.29	0.41	**0.41**	0.5	**2.0**	4.9		25	145	25	145	50	
Aroclor – 1242	1.0	0.29	0.41	**0.41**	0.5	**2.0**	4.9		30	145	30	145	50	
Aroclor – 1248	1.0	0.29	0.41	**0.41**	0.5	**2.0**	4.9		30	145	30	145	50	
Aroclor – 1254	1.0	0.29	0.41	**0.41**	0.5	**2.0**	4.9		30	145	30	145	50	
Surrogates: (same as 8081)														
DCB									30	135	30	135		
TCMX									25	140	25	140		

Criteria for the ratio (RD) of mean amount found to MDL: >1 but <5 and for MQL/MDL ratios (RQ) that are less than 3.

QC limit for RPD is from the USACE document, Appendix I, EM 200-1-3, February 1, 2001.

LCS (extended to the MS) %R limits are from the DoD QSM, Final Revision 2, June 2002.

The surrogate recovery limits shown for DCB and TCMX are from DoD QSM limits for 8081.

The following criteria will apply for marginal exceedances:

Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).

NOTE: Marginal exceedance concept is not applicable to PCBS analysis. Control limits and ME limits are identical.

NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.

TABLE 2

Soil/Sediment MDLs, LLRs, MQL and QC Limits for LCS/LCSD and MS/MSD Recoveries and RPDs Surrogate Recovery Limits

Polychlorinted Biphenyl (PCBs) Aroclors Analysis by GC/ECD

Analytical Management Laboratories

Preparation Method:	3550C		MDL Ext. Date:		6/20/01					Analyst:		RRH	
Analysis Method:	8082		MDL Anal. Date:		6/23/01					Matrix:		Soil	
	MDL	**MDL**	**MDL**	**MDL**	**LLR**	**MQL**	**Ratio (RQ)**			**LCS Limits**		**MS Limits**	**RPD**
Compound	Spike	DB-5	DB-1701	Report	Report	Report	QL/DL	Flag	Lower	Upper	Lower	Upper	Limit
	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg			**%R**	**%R**	**%R**	**%R**	%
Aroclor-1016	10	0.81	1.21	**1.21**	5	**33.3**	27.5		50	130	40	140	50
Aroclor-1260	10	1.00	1.86	**1.86**	5	**33.3**	17.9		50	130	40	140	50
Other Aroclors:													
(calculated results)													
Aroclor - 1221	10	0.91	1.54	**1.54**	5	**33.3**	21.7		50	130	40	140	50
Aroclor - 1232	10	0.91	1.54	**1.54**	5	**33.3**	21.7		50	130	40	140	50
Aroclor - 1242	10	0.91	1.54	**1.54**	5	**33.3**	21.7		50	130	40	140	50
Aroclor - 1248	10	0.91	1.54	**1.54**	5	**33.3**	21.7		50	130	40	140	50
Aroclor - 1254	10	0.91	1.54	**1.54**	5	**33.3**	21.7		50	130	40	140	50
Surrogates (same as 8081):													
DCB									55	130	55	130	
TCMX									70	125	70	125	

Criteria for the ratio (RD) of mean amount found to MDL: >1 but <10 for soil and for MQL/MDL ratios (RQ) that are less than 3.

QC limit for RPD is from the USACE document, Appendix I, EM 200-1-3, February 1, 2001.

LCS (extended to the MS) %R limits are from the DoD QSM, Final Revision 2, June 2002.

The surrogate recovery limits shown for DCB and TCMX are from DoD QSM limits for 8081.

The following criteria will apply for marginal exceedances:

Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).

NOTE: Marginal exceedance concept is not applicable to PCBS analysis. Control limits and ME limits are identical.

NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.

Table 3
List of Appendices

Appendix Number	Appendix Topic
1	Gas Chromatography Conditions
2	Retention Time Window Summary
3	Initial Calibration (ICAL) Summary (Form-6 equivalents) Instrument Printouts
4	Calibration Verification (ICV and CCV) Summary (Form-7 equivalents) Standard Chromatograms – Aroclor Patterns Instrument Printouts
5	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
6	Sample Result Worksheets (Form-10 equivalents)
7	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
8	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
9	Surrogate Recovery Reports (Form-2 equivalents)
10	Method Detection Limit (MDL) Study Summary
11	QC Association Forms (Form-4 equivalents) Sample Preparation Logsheets Instrument Run Log
12	Sample Reports (Form-1 equivalents)
13	USACE Method Quality Objectives for Method 8082 AFCEE MQOs for Method 8082 DoD QSM MQOs and Limits for percent recoveries

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices

STANDARD OPERATING PROCEDURES

No. O-TPHDiesel-RC

TOTAL PETROLEUM HYDROCARBONS (TPH) BY GC/FID EXTRACTABLE (EPH) OR DIESEL RANGE ORGANICS (DRO) FRACTION FLORIDA PETROLEUM RANGE ORGANICS (FL-PRO)

(EPA SW846-8000-8015-8100)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

TOTAL PETROLEUM HYDROCARBONS (TPH) BY GC/FID EXTRACTABLE (EPH) OR DIESEL RANGE ORGANICS (DRO) FRACTION FLORIDA PETROLEUM RANGE ORGANICS (FL-PRO)

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) is used to determine total extractable petroleum hydrocarbons (TPH-D, EPH, DRO, or FL-PRO) in a variety of matrices.

1.2 This SOP, which is applicable to nearly all types of samples, is based on guidance from EPA SW-846 Method 8015B/8015C/8015D, SW-846 Method 8000B/8000C, SW-846 Method 8100 and State TPH methods such as OA2, FL-PRO, and the Oklahoma DRO Method.

1.3 This method can be used to quantitate most TPH compounds that are extractable by methylene chloride and analyzed by gas chromatographic fused-silica capillary column coated with DB5.

1.4 Tables1 and 2 lists method detection limits (MDL), method quantitation limits (MQL) and reporting limits (RL), when applicable. Actual MQLs, MDLs and RLs will vary by the selected State method employed for DRO analysis.

1.5 The method or practical quantitation limits (MQLs or PQLs) for the analyte is instrument dependent and highly matrix dependent. MQLs will be proportionately higher for sample extracts and samples that require dilution to avoid saturation of the detector.

1.6 The words, SOP and method are used interchangeably in this document. This method is based upon the theories of gas chromatography/FID procedures. This method is restricted to experienced analysts in the use of these theories and skilled in the interpretation and use as a quantitative tool.

1.7 Several states have EPH or DRO methods that differ slightly from the procedures described in this document. The list of references included in this document should be used as additional guidance in meeting state requirements. Specific State methods/SOPs are included in the documents that are appended to this SOP.

1.8 DRO corresponds to the range of alkanes (and aliphatic and aromatic aromatic hydrocarbons in the range) from C_{10} to C_{28} and covering a boiling range of approximately 170°C to 430°C.

1.9 This SOP may be used for the identification of specific fuel types: Diesel, Jet Fuel, Hydraulic Fluid, Motor Oil, etc. However, the identification of specific fuel types may be complicated by environmental processes such as evaporation and biodegradation, presence of more than one type of fuel, etc.

2.0 SUMMARY OF METHOD

2.1 This method provides solvent extraction and gas chromatographic analytical conditions for the analysis of TPH compounds. Samples are extracted with methylene chloride, dried, concentrated to 1.0-mL, cleaned up when required, and analyzed by a gas chromatograph equipped with a flame ionization

detector (GC/FID). Detection and quantitation are based on FID response to a diesel or diesel component standard. Spiked samples are used to verify the chosen extraction procedure for each matrix.

3.0 INTERFERENCES

3.1 Interferences can be caused by a number of sources in the extraction and analyses stages. Quality control measures, such as field blanks, method blanks, solvent blanks and calibration blanks can be used to determine the interferences.

3.2 Contamination may occur when a high-concentration and a low concentration sample are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. A high concentrated sample should be followed by the analysis of solvent to check for possible cross contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph - A Hewlett Packard 5890 Series II GC with a HP FID detector and a HP 7673 autosampler is used. The system is complete with a temperature-programmable GC for splitless injection. The system includes all accessories, including syringes, analytical columns and gasses. The column flow rate remains constant throughout temperature program operation. The capillary column is directly coupled to the source.

4.2 Gas chromatographic column - A 30 meter x 0.32-mm ID capillary column coated with DB-5 (J&W Scientific) is employed. The film thickness is 0.25 um. An equivalent column may be used.

4.3 Data system - Hewlett Packard's Environquant is used to allow continuous acquisition and storage on machine-readable media of all chromatograms obtained throughout the duration of the GC program. The software allows searching any data file for plotting abundances versus time.

4.4 Microsyringes - 10, 25, 100, 250, 500 and 1000 µLs.

4.5 Balance - Analytical, 0.0001 grams and top loading, 0.1 grams.

4.6 Disposable pipettes - Pasteur

4.7 Test tubes - 10 ml to 15 ml, with Teflon lined screw caps.

4.8 Separatory funnels - 2000 ml

4.9 Kuderna-Danish (K-D) apparatus
- 10-ml concentrator tubes
- Evaporation flask (500 mls) attached to concentrator tubes
- Three ball macro Snyder column
- Clamps

4.10 Boiling Chips - solvent extracted

4.11 Heated water bath

4.12 Shaker

4.13 Tekmar Sonicator

4.14 Soxtec Extractors and Accessories

4.15 IKA automated Soxhlet Extractors and Accessories
4.16 Glass wool
4.17 Tablespoons
4.18 Spatulas

5.0 REAGENTS

5.1 All inorganic reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where available. Any other reagents must demonstrate high purity without lessening the accuracy of the determination.

5.2 Organic free reagent water - Use Type 1 reagent water as described in the AML SOP on reagent water. All references to water in this SOP will be assumed to be organic free reagent water.

5.3 Solvents

5.3.1 Methylene chloride - Pesticide Grade Quality

5.3.2 Acetone- Pesticide Grade Quality

5.4 Sodium sulfate - (granular, anhydrous), Na_2SO_4. Purify by heating to 400°C for four hours in a shallow tray.

5.5 Sulfuric acid (H_2SO_4), conc. (Fisher Scientific). Prepare 1:1 H_2SO_4 by slowly adding 50-mL of conc. H_2SO_4 to 50-mL of organic-free reagent water.

5.6 Sodium hydroxide (NaOH, 10N) - Prepare by dissolving 40-g of granular NaOH in 100-mL of organic-free reagent water.

5.7 Stock DRO Standard – Stock standard solutions are purchased from Ultra Scientific: Composite #2 Diesel Fuel Standard, catalog number RGO-616, at 50-mg/mL in methylene chloride. Transfer the standard to a screw-cap vial immediately after opening and store it at -10°C. Stock standards expire two years from the date of preparation or as stated by the vendor, whichever is earlier. Replace sooner if signs of degradation are observed.

5.8 Second Source DRO Standard - Purchase Diesel Fuel #2 stock standard from Restek (catalog number 31258-500) at a concentration of 50-mg/mL in methylene chloride. Transfer the standard to a screw-cap vial immediately after opening and store it at -10°C. Stock standards expire two years from the date of preparation or as stated by the vendor, whichever is earlier. Replace sooner if signs of degradation are observed. Use this stock standard to prepare initial calibration verification (ICV) solution.

5.9 Stock Surrogate Standard - The neat standard of p-Terphenyl (CAS # 92-94-4), the surrogate used for this analysis is purchased from Ultra Scientific (catalog number RAH-058). Dissolve the contents in 1-mL of methylene chloride to prepare a solution containing p-Terphenyl at a concentration of 100-mg/mL. Transfer the standard solution immediately to a screw-cap vial immediately after opening and store it at -10°C. Stock standards expire two years from the date of preparation or as stated by the vendor, whichever is earlier. Replace sooner if signs of degradation are observed.

5.10 Intermediate Surrogate Standard - Prepare an intermediate surrogate solution at 500-ug/mL that is used to prepare initial calibration solutions for the

surrogate by diluting 25-uL of the 100-mg/mL stock standard (Section 5.9) with methylene chloride to 5-mL in a volumetric flask. This standard expires one year from the date of preparation. Replace sooner if signs of degradation are observed.

5.11 Surrogate initial calibration solutions - These are prepared by diluting appropriate volumes of the intermediate solution (Section 5.10) to 1-mL with methylene chloride in a GC injection vial: (1) 500-ug/mL - no dilution; (2) 250-ug/mL - dilute 500-uL to 1-mL; (3) 100-ug/mL - dilute 200-uL to 1-mL; (4) 50-ug/mL - dilute 100-uL to 1-mL; and (5) 20-ug/mL - dilute 40-uL to 1-mL). Seal the vials with a septum cap using a crimping tool. These solutions are prepared fresh daily.

5.12 Surrogate Spiking Solution - Prepare 500-mL of the surrogate spiking solution at a concentration of 100-ug/mL in acetone by diluting 500-uL of the 100-mg/mL stock standard (Section 5.9) with acetone to 500-mL. Use 1-mL of this solution to spike each sample. This standard expires one year from the date of preparation. Replace sooner if signs of degradation are observed. NOTE: The quantity of spiking solution described above should be sufficient for 3-months of normal laboratory operation.

5.13 DRO Initial Calibration (ICAL) Solutions - These are prepared daily by diluting appropriate volumes of the DRO stock standard solution (Section 5.7) to 1-mL with methylene chloride in a GC injection vial with the exception of the highest level standard (50-mg/mL), which is the undiluted stock standard. Transfer the stock standard using pre-calibrated microliter syringes. Seal the vials immediately with a septum cap using a crimping tool. These solutions are prepared fresh daily.

5.13.1 50-mg/mL - Transfer 100-uL of the stock standard (Section 5.7) to a conical GC vial intended for small volumes.

5.13.2 10-mg/mL - Dilute 200-uL to 1-mL

5.13.3 5-mg/mL - Dilute 100-uL to 1-mL

5.13.4 1-mg/mL - Dilute 20-uL to 1-mL

5.13.5 0.6-mg/mL - Dilute 12-uL to 1-mL

5.13.6 0.3-mg/mL - Dilute 6-uL to 1-mL

5.14 Initial Calibration Verification (ICV) Solution - The ICV standard is prepared daily by diluting 20-uL of the second source DRO stock standard solution (Section 5.8) to 1-mL with methylene chloride in a GC injection vial. Transfer the stock standard using pre-calibrated microliter syringes. Seal the vial immediately with a septum cap using a crimping tool. This solution is prepared fresh daily.

5.15 Continuing Calibration Verification (CCV) Solution - The CCV standard is prepared daily by diluting 20-uL of the DRO stock standard solution (Section 5.7) to 1-mL with methylene chloride in a GC injection vial. Transfer the stock standard using pre-calibrated microliter syringes. Seal the vial immediately with a septum cap using a crimping tool. This solution is prepared fresh daily. Larger quantities of the solution may be prepared if multiple CCVs are needed for sample analysis.

5.16 DRO Spiking Solution for LCS/LCSD and MS/MSD samples - The Stock DRO standard solution (Section 5.7) is used to prepare the DRO spiking solution for spiking both MS and LCS samples and their duplicates (LCSD and MSD). The DRO spiking solution is prepared at a concentration of 1-mg/mL by diluting 1-mL of the 50-mg/mL DRO stock standard solution (Section 5.7) to 50-mL with acetone. Spike 1.0-mL of this solution for each LCS, LCSD, MS, and MSD sample analyzed. This standard expires one year from the date of preparation. Replace sooner if signs of degradation are observed. NOTE: The quantity of spiking solution described above should be sufficient for 3-months of normal laboratory operation.

5.17 DRO Hydrocarbon mixture - This standard (Ultra UST-200) is used to define the RT window (C_{10} and C_{28}) for DRO analysis and to establish the identity of resolved peaks in petroleum products. This standard contains several (C_{10}, C_{12}, C_{14}, etc., to C_{28}) hydrocarbons at a concentration of 2000-ug/mL in methylene chloride.

5.18 DRO RT Window and Baseline Standard - This standard is prepared from neat standards of the C_{10} or n-decane (Supelco cat # 442669, 1000-mg quantity) and C_{28} or n-octacosane (Supelco cat # 442696, 1000-mg quantity) and the intermediate surrogate standard (Section 5.10, p-Terphenyl at 500-ug/mL) as described below:

5.18.1 Dissolve the neat standard of n-decane (1000-mg) in 10-mL methylene chloride to prepare an n-hexane standard at a concentration of 100-mg/mL. This is a qualitative standard that expires in five years from the date of preparation. Replace sooner if signs of degradation are observed.

5.18.2 Dissolve the neat standard of n-octacosane (1000-mg) in 10-mL methylene chloride to prepare an n-octacosane standard at a concentration of 100-mg/mL. This is a qualitative standard that expires in five years from the date of preparation. Replace sooner if signs of degradation are observed.

5.18.3 Dilute 10-uL each of the 100-mg/mL n-hexane (section 5.18.1) and n-octacosane (Section 5.18.2) standards and 200-uL of the 500-ug/mL intermediate surrogate standard to 1-mL with methylene chloride. The resulting solution will contain the surrogate at the same concentration of the sample extracts (100-ug/mL) and the window compounds at a concentration of 1-mg/mL.

5.19 FL-PRO RT Window Standard - This hydrocarbon standard is purchased from Supelco.

6.0 SAMPLE PRESERVATION HANDLING & STORAGE

6.1 One liter amber glass bottles with Teflon lined screw caps may be used for collecting aqueous samples for DRO (TPH-D) analysis in accordance with

EPA SW-846 method 8015. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 2 bottles for each water sample to be collected. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. See project specific field sampling plan (FSP) for sample collection and preservation instructions. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. Although aqueous samples are collected without preservatives, samples for FL-PRO analysis should be collected in containers with the sulfuric acid preservative.

6.2 One 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples in accordance with EPA SW-846 Method 8015. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each solid sample. Quality control samples such as MS and MSD should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. These samples may be shared with samples collected for other analyses such as semivolatiles (8270C), chlorinated pesticides (8081A), PCBs (8082) and other pesticides (8140, etc.).

6.3 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction. Analytical data generated outside of the specified holding times must be considered to be minimum values only. The holding time for the extraction of aqueous samples is 7 days from the date of sample collection. The holding times for the extraction of soil, sediment and waste samples is 14 days from the date of sample collection. The holding time for the instrumental analysis of sample extracts is 40 days from the date of extraction.

7.0 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control contain a number of regulated compounds at high concentrations. The toxicity of these compounds present in such complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Aqueous and soil samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters after the solvent(s) have been allowed to evaporate in a hood.

9.0 EXTRACTION PROCEDURE

9.1 Preparation of waste samples

9.1.1 If the waste is soluble in methylene chloride, add approximately 1.00 gram and record the exact weight in the organic extraction logbook (See Appendix 7 for details), to a 15-ml culture tube. Add 1.0 ml of the surrogate spiking solution (Section 5.12) to each sample including QC samples (MB, LCS/LCSD, and MS/MSD). Add 1.0-mL of DRO spiking solution for LCS/LCSD and MS/MSD samples only (Section 5.16). Dilute the extract to 10.0-mL with methylene chloride and proceed with GC analysis.

9.1.2 If the waste is not soluble is methylene chloride, the sample must be extracted like a sediment/soil sample (See Section 9.2) using only 1.0 gram instead of 30 grams.

9.2 Preparation of Sediment and Soil Samples – A number of State TPH methods specify sonication for the extraction of soil samples. Procedures for extracting samples using automated Soxhlet methods are described in SOPs developed for other determinative methods (e.g. SVOCs, organochlorine pesticides, etc.), which should be consulted.

9.2.1 Add 30 grams of soil to a 500-ml beaker. Record the exact weight in the organic extraction logbook (See Appendix 7 for details). Add enough sodium sulfate (60-g is default) to dry the soil and mix well with a spoon. Add 1.0-mL of surrogate spiking solution (Section 5.12) to all the samples (including QC samples such as MB, LCS/LCSD, and MS/MSD). Spike LCS/LCSD samples and MS/MSD samples only with 1.0-mL of the DRO spiking solution (Section 5.16).

9.2.2 Add 100-mL of methylene chloride to the beaker and mix the contents with a spoon for 1 minute. Sonicate the soil/methylene chloride mixture for 3 minutes ensuring that the soil is free flowing and not in clumps. Set the output control knob at full power with the pulse mode switched on and the percentage cycle knob set at 50%. Do not use microtip probe. See AML SOP on sonicator tuning for tuning procedures that should be followed. Use Decant the extract through a funnel plugged with glass wool and covered

by sodium sulfate into a K-D apparatus. Repeat two more times, decanting each time.

9.2.3 Add two boiling chips to the K-D flask and attach a three ball Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract again reaches approximately 1.0 ml, remove the K-D flask and allow cooling for at least 10 minutes.

9.2.4 Concentrate the extract by nitrogen blow-down and adjust the volume to 1.0 mL as described in Section 9.2.5 and 9.2.6 respectively. Proceed with analysis by GC/FID.

9.2.5 Nitrogen blowdown technique - Place the concentrator tube in a warm water bath (approximately 35°C) and evaporate the solvent volume to the required level using a gentle stream of clean, dry nitrogen. The internal wall of the tube must be rinsed down several times with the appropriate solvent during the operation. During evaporation, the solvent level in the tube must be positioned to prevent water from condensing into the sample (i.e., the solvent level should be below the level of the water bath). Under normal operating conditions, the extract should not be allowed to become dry. When the apparent volume of liquid reaches 0.5-mL, remove the concentrator tube from the water bath. Adjust the final volume to 1.0-mL with methylene chloride.

9.2.6 Volume adjustment - Transfer the extract from the concentrator tube to a GC injector vial with 1-mL marking. Rinse the sides of the concentrator tube with small amounts of methylene chloride and transfer them to the GC vial. Adjust the volume to the mark using either Nitrogen blow-down or by adding additional methylene chloride. Seal the vial with a septum cap using a crimping tool.

9.2.7 Determine the percent solids by determining the initial weight of a sample aliquot (approximately 5-g) before and after drying the sample at 104°C. See AML SOP on percent solids for additional details. The percent solids determination is normally performed by analysts other than those performing the DRO analysis.

9.3 Preparation of Water Samples – Manual Liquid/Liquid Extraction

9.3.1 The aqueous samples are collected without any added preservatives. However, acid preservatives may be required for some client projects. Check the pH of the sample by withdrawing a few drop of sample using a Pasteur pipette onto a pH paper. If

the pH is <5 or >9 adjust pH using a few drops of 10N NaOH or 1:1 sulfuric acid, whichever is appropriate.

9.3.2 Mark the level of sample on the outside of the sample bottle and transfer the entire contents to a 2-L separatory funnel. If high concentrations are anticipated, a smaller volume of the sample diluted with de-ionized water to 1-Liter may be used. Add 1.0-mL of surrogate spiking solution (Section 5.12) to all the samples (including MB, LCS/LCSD, and MS/MSD).

9.3.3 An alternate procedure, which may be employed to measure out water samples, is described below. Using a 1-Liter graduated cylinder, measure out 1.00 liter ± 0.005 Liters (or lower volumes as low as 100-mL for high concentration samples) of the sample, and transfer it to a 2000-mL separatory funnel. Record the exact volume in the organic Extraction logbooks. This alternate procedure may be used to measure out lower sample volumes for the re-extraction of heavily contaminated samples with surrogate recoveries outside the QC limits, which are diluted out during analysis. This procedure may also be used to measure out smaller aliquots (e.g. 500-mL) of the sample for MS/MSD analysis, when additional sample containers were not collected for these analyses.

9.3.4 Using pH indicating paper (0-14) record the pH of the sample. This is accomplished by dipping the tip of a Pasteur pipette into the water and placing the droplets of water on the pH paper. Never place the pH paper into the water sample. NOTE: Check/Adjust the pH, if necessary, using either 50/50 sulfuric acid solution or 10N sodium hydroxide solution for specific State Methods.

9.3.5 Add 1.0- mL of the DRO spiking solution (Section 5.16) to the LCS/LCSD and MS/MSD sample only.

9.3.6 Rinse the bottle three times (NOTE: Use this procedure only when the whole sample is used for analysis) with about 5-mL of the extraction solvent (methylene chloride), transferring the rinsate each time to the separatory funnel.

9.3.7 Refill the sample bottle to the mark made with water, and then measure the volume of sample that was in the bottle, using a Class A 1-L graduated cylinder. Record the exact volume in the organic extraction logbook (See Appendix 7 for details) to a minimum of three significant numbers (e.g. 965-mL, 970-mL or 960-mL).

9.3.8 Add 60-mL of methylene chloride to the separatory funnel and shake for 2 minutes. Take care to vent the separatory funnel to relieve excess pressure. Allow the organic layer to separate from the water layer for a minimum of 10 minutes. Drain the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus. Repeat the extraction 2 more times using 60-mL aliquots of methylene chloride.

9.3.9 Add two boiling chips to the K-D flask and attach a three ball Snyder column. Pre-wet the Snyder column with 1 ml of

methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract again reaches approximately 1.0 ml, remove the K-D flask and allow to cool for at least 10 minutes.

9.3.10 Concentrate the extract by nitrogen blow-down (See Section 9.2.5) and adjust the final volume to 1.0-mL (See Section 9.2.6). Proceed with GC/FID analysis.

10.0 CALIBRATION PROCEDURES

10.1 Set up the gas chromatograph using conditions commonly used for semivolatile organics analysis (see Appendix 1 for details).

10.2 Screening and Pattern recognition

10.2.1 The calibration of DROs is markedly different from that of single component analytes. In particular, the response used for calibration must represent the entire area of the chromatogram within the retention time range for the fuel type. This includes the well-resolved peaks and the unresolved complex mixture that results in the "hump" in the chromatogram that is characteristic of the fuels. Although the resolved peaks are important for the identification of the specific fuel type, the area of the unresolved mixture constitutes a significant portion of the total response.

10.2.2 Pattern recognition - The hydrocarbon fuels are identified by GC pattern recognition. Distinguish hydrocarbon products from their pattern and the ranges of retention times that are characteristic for each type of fuel (see Appendix 1 for details).

10.2.3 Screening - Before injecting the initial calibration, the analyte of interest can be identified by injecting each hydrocarbon fuel at a medium level and then injecting the field samples. This will identify which hydrocarbon fuel is needed for the initial calibration. Once the particular hydrocarbon or hydrocarbons have been identified, the initial calibration can be analyzed. NOTE: Diesel #2 is the default petroleum product used for calibrating the instrument for most petroleum products. Use other standards (e.g. hydrocarbon mixtures, other petroleum products such as jet fuel, etc.) for specific projects or state methods requiring their use.

10.2.4 GC pattern recognition should be restricted to experienced analysts who have worked with TPH compounds.

10.2.5 For the analysis of DROs, sum the area of all resolved and unresolved peaks eluting between the retention times of C_{10} and C_{28}. Establish retention times (RT) and RT windows for the first

and last eluting compound by injecting a standard containing C-10 to C-28 alkanes (see Figure 1 in Appendix 1). See Section 7.6 of Method 8000B for additional guidance, if necessary.

10.2.5.1 Make three or more injections of the hydrocarbon mixture standard (Section 5.17 or 5.18.3) over the course of a 72-hour period. Record the retention times (min.) of C_{10} and C_{28} hydrocarbons to three decimal places. Calculate the mean (X) and standard deviation (SD) for each compound. If the observed standard deviation is <0.01 minute, use 0.01 as the default SD. Calculate RT window as X ± 3 * SD or ±3 * 0.01 minutes as applicable.

10.2.5.2 Establish the center of the retention time window (X) using the absolute retention time from the analysis of the RT window and baseline standard (Section 5.18.3) at the beginning of the analytical shift immediately before the analysis of CCV.

10.2.5.3 The RT range for DROs is calculated based on the lower limit of the RT window for the first eluting component (C_{10}) and the upper limit of the RT window for the last eluting component (C_{28}).

10.3 Inject the six DRO initial calibration solutions (Section 5.13) in the following order: 50-mg/mL, 10-mg/mL, 5-mg/mL, 1-mg/mL, 0.6-mg/mL, and 0.3-mg/mL. Inject the five surrogate initial calibration solutions (Section 5.11) using a similar order or include them in the ICAL standards.

10.4 Verify initial calibration by injecting a second source ICV standard (Section 5.14) immediately after the injection of ICAL standards (Section 10.3).

10.5 Tabulate the area response of the compound by integrating over the retention time window established (Section 10.2.5) for the petroleum product. The window defined by C-10 and C-28 should be used to report DRO. This area is generated by projecting a horizontal baseline between the retention times of C_{10} and C_{28} (see Figure 2 in Appendix 2 for details). Perform integration in the "integrate" mode by positioning the cursor on the baseline at the RT corresponding to the lower limit of the RT window for the first eluting component (C_{10}) and dragging the mouse over to a position on the baseline corresponding to the upper limit of the RT window for the last eluting component (C_{28}). Record the raw area and subtract background area as described in Section 10.5.1 to calculate corrected area response.

10.5.1 Background subtraction – See Section 11.11.12 of the SW-846 Method 8015D for additional guidance (when necessary) on this topic. Because the chromatographic conditions employed for DRO analysis can result in significant column bleed and a significant rise in the baseline, it is appropriate to perform a subtraction of the column bleed from the area of the DRO chromatogram. Additionally, the surrogate employed (p-Terphenyl) elutes close to C-24, which is within the DRO window. Therefore, the area

generated by projecting a horizontal baseline between the RTs of C_{10} and C_{28} will also include the area for the surrogate. Analyze an aliquot (same volume, e.g. 1-uL as that used for ICAL and samples) of the RT window and baseline standard (Section 5.18.3) during each 12-hour analytical shift immediately prior to the analysis of the CCV standard. The area of this chromatogram is measured in the same fashion as is used for samples (see Figure 1 in Appendix 1 for details), by projecting a horizontal baseline across the retention time range for DROs. This area is then subtracted from the area measured for the standard or sample and the difference in areas is used to calculate the DRO concentration.

10.6 Calculate the calibration factor (CF) for the compound using the following formula:

$$CF = \frac{\text{Adjusted GC Area in the RT window}}{\text{Concentration } (\mu g/mL)}$$

10.7 Calculate the average calibration factor (CF_m) and the standard deviation (SD) using the five or six points generated. The % RSD of the five calibration factors can be calculated using the following formula for each analyte:

$$\% \text{ RSD} = \frac{SD * 100}{CF_m}$$

An Excel spreadsheet may be used (see Appendix 3 for details) to calculate mean calibration factor and the %RSD. The calculated % RSD must not deviate by more then 20%. If the % RSD is greater than 20%, corrective action is required. Generally, regression methods (Section 10.8) are not required for GC/FID data due to the relatively high degree of linearity for the FID detector. When regression methods are also unacceptable, corrective action procedures, which must be performed would include the following: Cleaning the injection port, replacing liners and/or septum, cleaning the FID detector, making new standards or baking out the columns. The ICAL standards should be re-analyzed after correcting instrumental problems.

10.8 ICAL Options - If the % RSD of any detected compound is greater then 20%, use least square regression methods to calculate sample concentrations. For regression methods, the instrument response is treated as the dependent variable (y) and the standard amount is the independent variable (x). When "Cs" is used as "x", use "As*Cis/Ais" as "y". When "Cs/Cis" is used as "x", use "As/Ais" as "y". Do not include origin (0,0) as an extra calibration point.

10.8.1 Weighted least squares method is preferred for calculating concentrations more accurately at lower levels. A linear least squares equation (y = a*x + b) is preferred over non-linear calibration equations (y = a*x2 + b*x + c). The coefficient of determination (COD), coefficient of variation (r2) or correlation

coefficient (r) should be used to measure the goodness of fit. For regression models to be used for quantitative purposes, the COD, r2 or r must be greater than or equal to 0.99. A value of 1.00 indicates a perfect fit.

10.8.2 Corrective action may be required if the criteria for %RSD, r, r2, or COD are not met.

10.8.3 An initial calibration (ICAL) should be considered a single event process. Consult the QA office or check applicable EPA methods (e.g. SW-846 Method 8000C and 8081A) for additional guidance on appropriate corrective action.

10.8.4 Re-fitting of ICAL – The selected model for ICAL may be subjected to an additional re-fitting check to establish representativeness of the model for each compound in each ICAL standard. This is an optional procedure, which is recommended but not required since automated data processing procedures may not be able to accommodate it. Calculate the %D between the calculated amount (Cc) for each compound in each ICAL standard from the ICAL model and its expected value (Ce). Ideally, the %D ((Cc-Ce) * 100 /Ce) for each compound in ICAL standard should meet the QC criteria for %RSD. It is possible for one or more ICAL standards to have %D exceeding the %RSD criteria, while the %RSD for ICAL model is within QC limits. The re-fitting procedure may be used to identify ICAL standards that need re-analysis or dropped from the calculation of ICAL model when re-analysis was not performed.

10.8.5 Check and optimize instrument operating conditions including automated peak integration procedures for appropriateness in addressing the chromatographic needs of the specific compound with problems.

10.8.6 If the problem appears to be associated with just one of the ICAL standards, that one standard may be reanalyzed once immediately (must be within 8-hours) after the ICAL. Discard all the results from the original analysis of the standard in question and replace it with the results from re-analysis.

10.8.7 When additional calibration standards (six or seven instead of five) are analyzed, narrowing the range of concentrations to be used for determining the goodness of fit is an acceptable procedure. Five or more standards should remain for the calculation of RSD or linear regression for each compound after one or more points are removed from the ICAL. More points (6 for second order and 7 for third order) may be needed for non-linear regression. It is generally prohibited to remove data points from within a calibration range, while still retaining the extreme ends of the calibration range. Do not discard a middle point unless it appears to have an obvious problem and six or more standards were analyzed for ICAL and re-analysis of the affected standard was not

performed within the required time limits. Changes to the upper end of the calibration range will affect the need to dilute samples above the range. A change to the lower range is not recommended since it will affect the method quantitation limit (MQL) used to report data. The default MQLs, which are based on the lowest ICAL concentration are stored in the LIMS and project-specific changes are not practical.

10.8.8 The refitting procedures may lead to failed %Ds for upper level calibration standards for average response factor methods. The analyst may take alternate corrective actions such as the analysis of sample extracts under dilution for the affected compounds.

10.8.9 The refitting procedures may lead to failed %Ds for lower level calibration standards for regression methods. The analyst may take appropriate corrective action such as flagging the sample result as an estimated amount ("J" flag) at the lower level encountered in the sample.

10.9 The term "calibration verification" (CALV) is used to describe initial verification (ICV) of the initial calibration (ICAL) using second-source standards and subsequent continuing verifications (CCVs) using same-source standards. The response factors calculated during CALV are not used for sample quantitation since CALV standards are not used as "continuing calibration" standards permitted in some of the EPA methods.

10.10 Initial Calibration Verification (ICV) - Analysis of a second source ICV standard is required before any samples can be analyzed for USACE projects. Since second source standards are not employed for the analysis of LCS/LCSD and MS/MSD samples, ICV analysis is applicable for all projects. Calculate the percent difference (%D) or %Drift for the observed response factor in ICV from the mean RF as described for CCV. The %D or %drift should not exceed ±15% for each target compound. Document calibration verification in instrument printouts as shown for CCVs. Check the ICAL and ICV standards if it is not achievable for all the target compounds without any exception. Use professional judgment for non-USACE projects and for compounds that are not contaminants of concern (COC) for USACE projects. The ICV may be used as the beginning CCV for sample analysis. If the above criteria are met, the analysis for analytical samples can begin.

10.11 The calibration must be verified every 10 instrument injections by injecting a CCV solution (Section 5.15). If the % drift for the calibration factor from the mean calibration factor obtained during initial calibration is less than ±15%, the initial calibration is assumed to be valid. If the criterion is not met (i.e., >15%), corrective action must be taken. If the %D is exceeded, a new five point calibration must be generated if other corrective action measures (see Section 10.7) fails to restore the machine to the original condition of the machine during the analysis of ICAL. This criterion must be met before any quantitative analysis for samples can begin.

10.12 Document calibration verification on spreadsheets (see Appendix 3 for details). The calculation for the % Difference (%D) between the initial calibration (CF_m) and the calibration check (CF_v) is shown below:

$$\%D = \frac{(CF_v - CF_m) * 100}{CF_m}$$

where:

CF_m = Mean Calibration Factor from initial calibration
CF_v = Calibration factor for ICV or CCV

11.0 ANALYSIS OF EXTRACTED SAMPLES

11.1 Once the samples have been extracted and screened, the extracts can be quantitatively analyzed.

11.2 If the response for any quantitation extract exceeds the initial calibration curve range of the GC system, extract dilutions must take place. The diluted extract must be reanalyzed with a valid initial calibration.

11.3 Water Samples

11.3.1 The following formula is used to calculate any detected analytes in a water sample:

$$\text{Concentration } (\mu g/L) = \frac{A_s * V_e * D}{CF_m * V_s}$$

Where:

A_s = Adjusted GC Area for the Sample
CF_m = Mean ICAL CF for the Hydrocarbon (mL/µg)
V_e = Final volume of the extract (mL)
D = Dilution Factor for instrumental analysis
V_s = Sample Volume (L)

11.4 Soil Samples

$$\text{Concentration } (\mu g/Kg) = \frac{A_s * V_e * D * 100}{CF_m * W_s * (\%S)}$$

Where:

A_s = Adjusted GC Area for sample
CF_m = Mean ICAL calibration factor (mL/µg)
V_e = Volume of the sample extract (mL)
D = Dilution Factor for instrumental analysis
W_s = Sample Wet Weight (Kg)
%S = % Solids

12.0 QUALITY CONTROL

12.1 Analytical data, which includes calibration data, sample preparation logs, sample data and quality control data should be gathered prior to review. A checklist (see Appendix 4 for details) should be used to document verification of calculations and adherence to method procedures. Exceptions should be documented. Samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on these checklists. Summarize sample results including those for QC samples such as MB, LCS, LCSD, MS, and MSD as TPH-Diesel or DRO on Excel spreadsheets (see Appendix 3 for details).

12.2 Surrogate recovery should be within QC limits for samples, spikes and blanks. The laboratory QC limits for interference-free matrix (MB, LCS, and LCSD) are tighter (50% to 130%) than the laboratory limits (40-140%) for project samples. Report surrogate recoveries in Form-2 equivalents (see Appendix 8 for details). Flag recoveries that are outside the QC limits with an asterisk (*). If re-extraction and/or reanalysis has been performed, and the surrogate recoveries for reanalysis are within QC limits, report data based on acceptable surrogate recoveries. If the surrogate recoveries are outside the QC limits for the reanalysis, report data for both analyses with surrogate recovery flags.

12.3 Method blanks (MB) should be free of target compounds. Report DRO in the MB at levels greater than the MDL (see Appendix 9 for details) or the reporting limit (RL) as appropriate using J qualifiers if the levels are less than the method quantitation limit (MQL) in Form-1 equivalents (see Appendix 10 for details). Report QC association of samples in Form-4 equivalents (see Appendix 7 for details). The action level for method blank is one half of the MQL. See Tables 1 and 2 for a list of MDLs and MQLs. Samples associated with the method blank containing TPH-Diesel at levels greater than one half the MQL will require re-extraction and reanalysis unless other criteria acceptable to the client are used. Since method blanks may be contaminated when they are associated with samples containing high levels of target compounds, only samples that contain less than one tenth of the MB amount may need to be re-extracted and reanalyzed.

12.4 LCS and/or LCSD (interference-free matrices) recoveries should be within QC limits (50-130%) for soil and water matrices, which are tighter than those for actual samples (MS/MSD). Flag recoveries that are outside the QC limits with an asterisk (*). See Tables 1 and 2 for QC limits. Report LCS/LCSD results in Form-1 equivalents and recoveries/RPD in Form-3 equivalents (see Appendix 5 for details). See AML SOP (See Section 14 for SOP references) on control charting for additional details. Contact supervisor for appropriate corrective action if GRO recoveries are outside the QC limits. Data qualification, re-extraction, and/or reanalysis of associated samples may be necessary depending on project requirements, turnaround time and other constraints.

12.5 MS recoveries should be within QC limits (40-140%) for soil and water matrices. Flag recoveries that are outside the QC limits with an asterisk (*). See Tables 1 and 2 for QC limits. Report MS/MSD results in Form-1 equivalents and recoveries/RPD in Form-3 equivalents (see Appendix 6 for details). See AML SOP (See Section 14 for SOP references) on control charting for additional details. Contact supervisor for appropriate corrective action if DRO recoveries are outside the QC limits. Data qualification, re-extraction, and/or reanalysis of associated samples may be necessary depending on project requirements, turnaround time and other constraints.

12.6 Duplicate analyses should be performed with each batch of samples analyzed. Duplicate analyses may include one or more of the following: LCS duplicates, sample duplicates, or MS duplicates. The relative percent difference (RPD) for duplicates should be within QC limits (<50% for soil and water).

12.7 Every effort must be made to identify the petroleum product found in the sample. Calculate DRO as compounds that are detected in the retention time window defined by C-10 and C-28. Use other windows as appropriate for products such as mineral spirits, kerosene, lubrication oil, hydraulic fluid, fuel oil, etc. Report DRO that is present at levels greater than the reporting limit (RL) using "J" qualifiers if the levels are less than PQL. GRO detected in the associated MB should also be qualified with a "B" code. If the concentration of DRO exceeds the upper limit of calibration (ICAL), flag the result with an estimated or "E" flag. The extracts may need to be diluted and reanalyzed until the concentrations are within the calibration limits for normal turnaround analyses. For rapid turnaround analyses sample results may be reported with "E" qualifiers if sufficient time is not available for reanalysis. Sample results should be reported in Form-1 equivalents (see Appendix 10 for details).

13.0 DATA VERIFICATION AND VALIDATION - This section will provide guidance and procedures to assess if data produced using this SOP is valid and legally defensible. Document review and comments on the checklist (See Appendix 4 for details). Verify that sample preparation logs are complete and the instrument run logs are available for review (See Appendix 7 for details). Anomalies and exceptions should be documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on the checklists. Army (USACE) and Air Force (AFCEE) projects may have special requirements (See Appendix 11) that will require extra attention. A simple, universal procedure for evaluating and choosing appropriate corrective action doe not exist to deal with situations when one or more QC parameters are outside the acceptance window. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints.

13.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) or air force (AFCEE) projects may have additional requirements that are related to USACE laboratory validation (CX), USACE District requirements, government contract requirements, specific project requirements or Architect/Engineer (A/E) requirements. Requirements may also be from the proposed implementation of the Department of Defense Quality Systems Manual (QSM) for Environmental Laboratories (Final Revision 2, June 2002), that will govern analyses performed for Army, Navy, Air Force, and the Marine Corps. Contact supervisor for specific client requirements (see Appendix 11 for details).

13.1.1 The USACE and/or AFCEE requirements (see Appendix 11 for details) and elsewhere in this SOP are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may or may not be attainable for routine sample analysis. Since MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

13.1.2 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality objectives and/or other QC requirements. Exceptions and data qualifications necessary should be documented on the checklists and eventually on case narratives.

13.1.3 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist, perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete.

13.1.4 For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete.

13.1.5 For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director will review the checklists (initial/date as necessary), prepare case

narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

13.2 HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

13.2.1 WATER - The holding time for extraction is 7 days from the sampling date.

13.2.2 SOIL – A 14 day holding is used for the extraction of soil and waste samples.

13.2.3 The holding time for instrumental analysis of sample extracts (soil and water) is 40-days from the date of extraction.

13.2.4 Holding times are established by comparing the chain of custody sampling date with the date of the injection after purging.

13.2.5 ACTION – If holding times have been exceeded, contact the client. All positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or invalid (I).

13.3 INITIAL CALIBRATION – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the GC/FID is capable of producing data within a range that is linear. For USACE and AFCEE projects, the ICAL should be verified using a second source (ICV) calibration verification solution (see Appendix 11 for details).

13.3.1 %RSD for DRO – Check to see that the 20% criteria is met. If it is not met, check to see if linear regression can be used. The limit for "r" used in linear regression is 0.99. Otherwise, correct the problem and repeat the initial calibration process.

13.3.2 ACTION – Positive results for compounds not meeting the criteria may need to be qualified as estimated (J) by the user (client). Report detects and non-detects without qualifiers and mention these outliners in the case narrative.

13.4 CALIBRATION VERIFICATION – The calibration verification consists of two parts: (a) Initial calibration verification (ICV), which is performed using a second source standard to check on the ICAL standard; and (b) Continuing calibration verification (CCV), which is used to check that the instrument is operating under the linear range of the initial calibration. The CCV is also used to check the stability of the machine every 12 hours.

13.4.1 Check to see if the %D (%drift or %difference) for DRO in ICV and/or CCV is 15% or less.

13.4.2 ACTION – If the %D is >15%, check the ICAL standard and the ICV standard. Replace the ICV standard with a fresh standard and repeat ICV. If the problem is not solved, replace the ICAL standards. Replace the intermediate and stock standards, if necessary, if the problem is not solved.

13.4.3 ACTION - If the %D is >15% for CCV, correct the problem as described for ICV and repeat the calibration verification process.

13.4.4 ACTION - If the standard analyzed after a group of samples exhibits a response that is above the 15% limit (>115%) and DRO was not detected in the samples, then those samples do not need to be re-analyzed, as the verification standard has demonstrated that gasoline would have been detected had it been in the samples. If DRO had been detected, then it would be necessary to re-analyze the samples with an acceptable verification standard or new ICAL.

13.4.5 ACTION - If DRO was not detected in the samples, but the verification standard was below the ICAL response by more than 15% (<85%), then re-analysis of the samples would be necessary.

13.4.6 ACTION - If CCV failures are noted, positive results for compounds not meeting the criteria may be qualified as estimated (J) by the user (client). Report non-detects without qualifiers and mention these outliers in the case narrative.

13.5 METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample handling and processing procedures, from the field to the laboratory. According to SW-846 the MB result should be less than the MDL for the analyte, less than 5% of the regulatory limit associated with an analyte, or less than 5% of the sample result for the analyte, whichever is greater. Sample data qualification based on all the blank results (laboratory MB, equipment blank, and other field blanks) is performed by the user (client).

13.5.1 Method blanks should be free of all target analytes and other interfering non-target analytes. To be acceptable, blank results should not exceed one half of the applicable PQL.

13.5.2 Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples. If levels greater than one half the PQLs are present, inspect and correct the problem. Reanalyze all associated samples. Contact client if other criteria are applicable. The only exception may be quick turn around projects.

13.5.3 ACTION – All positive results are reported with a “B” code, if contaminants are found in the method blank.

13.5.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, then no action is taken.

13.5.5 ACTION – Any compound detected in the sample which were also detected in associated method blank at levels greater than one half

the PQLs should be re-extracted unless other client criteria are applicable.

13.5.6 ACTION – If contaminants resulting in GC/FID peak saturation are present, all affected compounds are qualified as invalid (I).

13.6 SURROGATE RECOVERIES – Surrogates or system monitoring compounds are different structurally, but similar chemically to classes of target compounds. These surrogate compounds are added just prior to extraction of the samples and the percent recoveries of the surrogates are calculated and evaluated.

13.6.1 Verify that the surrogate recoveries are within QC limits by comparing QC limits (40% to 140% for samples and 50% to 130% for MB, LCS, and LCSD). Flag outliers with an asterisk (*).

13.6.2 ACTION – When surrogate recoveries are outside control limits the sample must be re-analyzed. Reanalysis may be performed under dilution if original analysis should indicate high levels of target and non-target analytes. If surrogate recoveries are still outside the limits, report both sets of data and described the problem in the case narrative.

13.7 LABORATORY CONTROL STANDARD (LCS) – A LCS analysis is used to evaluate method competence in the absence of matrix effects. The LCS should be from a second source standard. The LCS may provide a mechanism to check method efficiency for target compounds without the sample matrix as a factor.

13.7.1 Check the results for the LCS recoveries to see if the results are within the laboratory QC limits (50-130% for soil and water). Flag outliers with an asterisk (*). Since this is a single analyte procedure, there is no allowance for marginal exceedances.

13.7.2 ACTION –If significant deviations are noticed, check retention windows, integration parameters, chromatographic baseline, and calculations. Correct any observed problem. Reanalysis of the LCS and associated samples may be necessary depending on client and project requirements. Re-extraction may also be necessary if reanalysis does not solve the observed problem. Check with the project manager about such project requirements. Professional judgment may be used by the user (client) in evaluating sample data qualification options in conjunction with other QC data for the project.

13.8 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike data are used to evaluate the precision and accuracy of the analytical method on project samples. The percent recoveries (%R) are calculated for MS and MSD and the relative percent difference (RPD) is calculated for duplicate analyses, which includes MS duplicates. Since this is a single analyte procedure, there is no allowance for marginal exceedances.

13.8.1 Check the results for the MS/MSD recoveries against laboratory QC limits (40-140% for %R). Flag outliers with an asterisk (*).

13.8.2 Perform one manual calculation using raw data and extraction sheet information.

13.8.3 Check the observed RPD for duplicates against laboratory QC limits (<50% for soil and water). Flag outliers with an asterisk (*).

13.8.4 NOTE – MS data evaluation is more complex than method blank or LCS data evaluation since they are complicated by matrix effects in addition to sample preparation and analysis errors. The heterogeneity of grab soil samples, and potential non-representativeness of sequentially collected water samples, etc. further complicate the evaluation since it is assumed that the native concentrations are constant in triplicate analyses. In addition, concentrations of the target analytes in the sample can also far exceed spike amounts. If the native concentration of target analytes in the sample chosen for spiking is high relative to the spiking concentration, the differences in the native concentration between the unspiked sample and the spiked samples may also become very significant.

13.8.5 NOTE - If no project samples are collected for MS analysis, samples from another project used for MS analysis may not be representative of the project samples. When project samples (e.g. surface soil) are chosen, their MS recovery characteristics may be different from those of other samples (e.g. sub-surface soil) in the same batch.

13.8.6 ACTION - The results of the MS are evaluated, in conjunction with other QC information, to determine the effect of the matrix on the bias of the analysis. The supervisor should be consulted if this is not achievable. Matrix spike recoveries that fail to meet the appropriate acceptance criteria may indicate that a potential matrix effect is present. A review of the MSD result, if available, may confirm the matrix effect, if it is in the same direction and same order of magnitude. Check the LCS and/or LCSD recoveries for affected compounds to rule out laboratory problems.

13.8.7 ACTION - If the sample chosen for MS/MSD is inappropriate, notify the client. When additional payments are authorized for re-analysis, re-extract and/or reanalyze the MS/MSD samples after employing cleanup procedures (if applicable), dilution techniques to minimize matrix interference, or using other project samples that appear to be clean, when sufficient quantities are available. Re-spike the sample at a higher level (e.g., at approximately two to four times the sample concentration), then reanalyze the sample based on project-specific requirements. It is the responsibility of the client at all times to select appropriate samples for MS analysis, make payments for additional analyses, and to offer necessary guidance as needed to meet their data quality objectives.

13.8.8 ACTION - If the matrix effect cannot be resolved, discuss the situation in the case narrative. No action may be taken by the laboratory to qualify associated sample data based on MS/MSD recovery data alone. Professional judgment may be used by the user (client) in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

13.9 TARGET COMPOUND IDENTIFICATION AND DOCUMENTATION – Criteria for petroleum product identification are to minimize the number of erroneous positive or negative identifications of petroleum products. Document sample analyses by generating instrument printouts and Excel spreadsheets (see Appendix 2 and 3 for details). Document review in a checklist (see Appendix 4 for details). Compare observed results for each sample with adjusted MDLs, MQLs and associated method blank results to determine appropriate data qualification on sample reports (Form-1 equivalents). See AML SOP on MDLs and control charting (Section 14) for more details. Prepare a QC Association form (Form-4 equivalent) for samples (includes MS and MSD samples) that are associated with batch QC (MB, LCS, and/or LCSD).

13.10 TARGET COMPOUND REPORTING AND DATA QUALIFICATION - Report analytes that were detected at concentrations that are less than the MDL and/or the lowest level of reporting (LLR) as not detected with a "U" flag. Report analytes that were detected at concentrations that are greater than the MDL and/or the LLR but lower than the MQL as estimated with a "J" qualifier. Report analytes that were detected at concentrations that are greater than the upper limit of calibration (adjusted for sample weight/volume, dilutions, and %moisture) as estimated with an "E" qualifier to indicate that calibration range has been exceeded. Reanalyze samples that contain "E" flagged data under dilution to yield data without such flags. Provide both sets of results for use by the client.

14.0 REFERENCES

14.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8015B, Volatile and Semivolatile Organic Compounds by Gas Chromatography/Flame Ionization Detector (GC/FID), December 1996.

14.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8015C, Volatile and Semivolatile Organic Compounds by Gas Chromatography/Flame Ionization Detector (GC/FID), Revision 3.

14.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8015D, Nonhalogenated Organics using GC/FID, Revision 4, June 2003.

14.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.
14.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.
14.6 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3500B, Organic Extraction and Sample Preparation, December 1996.
14.7 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3520C, Continuous Liquid-Liquid Extraction, December 1996.
14.8 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3510C, Separatory Funnel Liquid-Liquid Extraction, December 1996.
14.9 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3540C, Soxhlet Extraction, December 1996.
14.10 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3541, Automated Soxhlet Extraction, December 1996.
14.11 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3545, Pressurized Fluid Extraction, December 1996.
14.12 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3550B, Ultrasonic Extraction, December 1996.
14.13 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3580A, Waste Dilution, December 1996.
14.14 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3585, Waste Dilution for Volatile Organics, December 1996.
14.15 Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, EPA, 40 CFR Part 136, Method 625: Base Neutral and Acid Compounds, October 1984.
14.16 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.
14.17 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.
14.18 AML Standard Operating Procedures, Q-Sonicator-RD.
14.19 AML Standard Operating Procedures, Q-Water-RC.
14.20 AML Standard Operating Procedures, Q-WasteMgmt-RD.
14.21 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
14.22 AML Standard Operating Procedures, Q-Controlcharts-RC.

14.23 Determination of total purgeable (diesel range) petroleum hydrocarbons (OA-2), State of Iowa, 1991.

14.24 MADEP, "Method for the Determination of Extractable Petroleum Hydrocarbons (EPH), MADEP-EPH-98-1, January 1998.

14.25 Modified DRO - Method for defining Diesel Range Organics, Wisconsin Department of Natural Resources, PUBL-SW-141, 1992.

14.26 NWTPH-Dx - Semivolatile Petroleum Products Method for Soil and Water, Northwest (Oregon and Washington) Total Petroleum Hydrocarbon Analytical Methods, State of Oregon DEQ lab, December 1996.

14.27 NWTPH-HCID - Hydrocarbon Identification Method for Soil and Water, Northwest (Oregon and Washington) Total Petroleum Hydrocarbon Analytical Methods, State of Oregon DEQ lab, December 1996.

14.28 Petroleum Range Organics, FL-PRO, State of Florida.

14.29 Diesel Range Organics (DRO), Oklahoma Department of Environmental Quality, Methods 8000/8100(modified), 10/22/97, Revision 4.1.

Table 1
Aqueous Sample MDL, MQL, and QC Limits for LCS and MS

Total Petroleum Hydrocarbons (TPH) Analysis by GC/FID

Analytical Management Laboratories

Matrix: Water	**Sample Preparation**		**Sample Analysis**			**Technique**
Parameter	Method No.		Method No.	Date	State UST	
DRO	3510C		8015B		OA2	Extraction & GC/FID

	Spike Amount	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**		**LCS Limits %R**		**MS Limits %R**		**RPD Limit**
Parameter	(mg/L)	**Mg/L B**	mg/L	**mg/L A**	RD= B/A	flag	**mg/L C**	RQ= C/A	flag	Lower	Upper	Lower	Upper	%
DRO	0.06	0.039	0.006	**0.017**	2.3		0.3	17.2		50	130	40	140	50

DRO = Diesel Range Hydrocarbons or TPH-Diesel (Extractables).

OA2 is a State (Iowa, Kansas, and Missouri) UST method based on SW 8015 for DRO.

Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 10 for multicomponent analytes.

Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.

LCS and MS %R limits are laboratory limits for multicomponent analytes with guidance from USACE EM200-1-3, Appendix I.

Table 2
Soil/Sediment Sample MDL, MQL, and QC Limits for LCS and MS

Total Petroleum Hydrocarbons (TPH) Analysis by GC/FID

Analytical Management Laboratories

Matrix: Soil	**Sample Preparation**		**Sample Analysis**			**Technique**
Parameter	Method No.		Method No.		State UST	
DRO	3550B		8015B		OA2	Extraction & GC/FID

	Spike Amount	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**		**LCS Limits %R**		**MS Limits %R**		**RPD Limit**
Parameter	(mg/kg)	**mg/kg B**	mg/kg	**mg/kg A**	RD=B/A	flag	**mg/kg C**	RQ=C/A	flag	Lower	Upper	Lower	Upper	%
DRO	10	8.2	0.64	**2.01**	4.1		10	5.0		50	130	40	140	50

DRO = Diesel Range Hydrocarbons or TPH-Diesel (Extractables).

OA2 is a State (Iowa, Kansas, and Missouri) UST method based on SW 8015 for DRO.

OA1 and OA2 are State (Iowa, Kansas, and Missouri) UST methods based on SW 8015 for GRO and DRO respectively.

Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 10 for multicomponent analytes.

Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.

LCS and MS %R limits are laboratory limits for multicomponent analytes with guidance from USACE EM200-1-3, Appendix I.

Table 3
List of Appendices

Appendix Number	Appendix Topic
1	Chromatogram for RT Window Compounds (Figure 1) for DRO Retention Time (RT) Ranges for Diesel Range Organics Determination of Area Response (Figure 2) for DRO Chromatograms for Common Petroleum Products
2	Instrument Printouts Quantitation Reports and Chromatograms Peak areas for DRO
3	Initial Calibration (ICAL) Summary (Form-6 equivalent benchsheets) Calibration Verification Summary (Form-7 equivalent benchsheets) Sample Results Summary (Form-1 equivalent benchsheets) Surrogate, LCS and MS Summary (Form-2 and 3 equivalent benchsheets)
4	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
5	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
6	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
7	QC Association Forms (Form-4 equivalents) Sample Preparation Logsheets Instrument Run Log
8	Surrogate Recovery Reports (Form-2 equivalents)
9	Method Detection Limit (MDL) Study Summary
10	Sample Reports (Form-1 equivalents)
11	Client Method Quality Objectives for Method 8015 mod. (AFCEE, USACE, DoD QSM, etc.)

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. O-TPHGasoline-RC

TOTAL PETROLEUM HYDROCARBONS (GASOLINE FRACTION) BY GAS CHROMATOGRAPHY/FID (GC/FID) (OA-1, GRO, and EPA SW846-8015D mod.)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure SOP) is used to determine the total petroleum hydrocarbon compound, gasoline range, in a variety of matrices. This SOP, which is based on EPA SW-846 Method 8015B (including more recent Revisions C and D) and several State (e.g. Iowa OA-1, Wisconsin GRO, MADEP-VPH, etc.) undergraduate storage tank (UST) methods is applicable to nearly all types of samples.

1.2 Gasoline Range Hydrocarbons (GRO) is defined in SW-846 Method 8015D as the range of alkanes (or hydrocarbon compounds) from C_6 to C_{10} and covering a boiling point range of approximately 60°C to 170°C.

1.3 The method detection limit for this analyte, which is also known as gasoline range organics (GRO), is 0.15-mg/L for water and 0.25-mg/kg for soil.

1.4 The method quantitation limit (MQL) for gasoline in water is 0.5 to 1.0-mg/L. For soil matrices the MQL is approximately 0.5- to 1.0-mg/kg. The MQLs will be proportionately higher for sample extracts and samples that require dilution. The MQLs can be lower for aqueous samples when 25-mL purge volumes (default is 5-mL) are employed.

1.5 The instrument employed at Analytical Management Laboratories (AML) is a Hewlett Packard 5890 GC equipped with a flame ionization (FID) detector, which can detect and quantitate aliphatic and aromatic hydrocarbons and a photoionization detector (PID), which is more specific for aromatic hydrocarbons.

1.6 The terms, SOP, procedure, and method are used interchangeably in this document. This method is based upon the theories of purge-and trap and gas chromatography/FID procedures. This method is restricted to experienced analytes in the use of these theories and skilled in the interpretation of chromatograms and their use as a quantitative tool.

1.7 With minor modifications (for aromatic hydrocarbon determination), the procedures described in this SOP may also be used to determine volatile petroleum hydrocarbons (VPH) in accordance with the Massachusetts Department of Environmental Protection (MADEP) method. Details of determining MADEP-VPH are described in other method documents.

2.0 SUMMARY OF METHOD

2.1 The gasoline compound is introduced into the gas chromatograph by the purge-and-trap method. Purged sample components are trapped in a tube containing suitable sorbent materials. When purging is complete, the sorbent tube is heated and backflushed with helium to desorb trapped sample components. The analyte is desorbed directly to a large bore capillary column before being flash evaporated to a narrow bore capillary for analysis. The column is temperature programmed to separate the analytes, which are then detected with a FID detector interfaced to the gas chromatograph.

2.2 Sometimes, due to high levels of target analytes, it is necessary to extract the TPH with a suitable solvent such as methanol. Some state methods (e.g. MADEP-VPH) require sample collection using the methanol preservative. When procedures based on EPA SW-846 Method 5035A are employed, sample collection may involve acid preservation with sodium bisulfate and methanol preservation/extraction for high concentration samples. A portion of the methanol solution is combined with organic-free reagent water in the purge chamber. It is then analyzed by purge-and-trap GC/FID following the normal water method.

2.3 Identification of the target analyte is accomplished by comparing the GC pattern with authentic standards. Quantitation is accomplished by comparing the response of the total chromatogram using the external standard method with a five-point calibration curve.

3.0 INTERFERENCES

3.1 Impurities associated with common laboratory solvents (e.g. non-purge and trap grade methanol), the widespread use of petroleum fuel and other impurities from volatile materials are a major source of contamination. Analyses of blanks and calibration data should give insight to the source of the contamination. Corrective action should be enforced when dealing with a contamination. Subtracting blank value from samples is not permitted. An explanation of any blank contamination will accompany the final report in the case narrative.

3.2 Interfering contamination may occur when low levels of the target analyte are found immediately following the analysis of a sample containing high levels of the target analyte. All purging apparatuses and syringes are rinsed after each sample analysis regardless or carry-over. All glassware is cleaned, heated to 105° C and allowed to cool to room temperature prior to analysis. Additional blanks may be analyzed to check for sample contamination. Screening by dilution is also employed in many instances. Generally, as a preventive maintenance measure, the purge-and-trap system is baked out at 250° C along with each individual sampler position.

3.3 A trip blank (when collected by the clients) is analyzed to check for contamination during the shipment and storage procedures. A trip blank is usually prepared by the laboratory using organic free reagent water and then delivered to the field samplers, who then keep the trip blank with the actual field samples.

3.4 The oven temperature program is designed to bake out semi-volatile hydrocarbons that may have volatilized during the purge-and-trap analysis.

4.0 APPARATUS AND MATIERIAL

4.1 Purge-and-trap device - A Tekmar 3000 Concentrator, equipped with a Archon autosampler.

4.2 Gas Chromatograph - A Hewlett Packard 5890 Series II GC, equipped with PID and FID detectors in tandem. The instrument is also equipped with a HP 5970 mass spectrometer for analyte confirmation.

4.3 The P&T-GC/FID system is complete with a temperature-programmable GC for splitless injection or interface to the Tekmar. The system includes all accessories, including syringes, analytical columns and gases. The column flow rate remains constant throughout desorption and temperature program operation. The capillary column is directly coupled to the source.

4.4 Gas chromatographic column - A 60 meter x 0.25 mm ID capillary column coated with DB-624 (J&W Scientific) is employed. The film thickness is 1.8 um. An equivalent column may be used.

4.5 GC/FID Interface is directly coupled by inserting the column into the FID detector.

4.6 Data system - Hewlett Packard's Environquant is used to allow continuous acquisition and storage on machine-readable media of all chromatograms obtained throughout the duration of the GC program.

4.7 Microsyringes - 10, 25, 100, 250, 500 and 1000 uLs.

4.8 Syringes - 5 mL syringe with Luer ends.

4.9 Balance - Analytical, 0.0001 grams

4.10 Disposable pipettes - Pasteur

4.11 Volumetric Flasks, class A - 10 mL and 100 mL with ground glass stoppers.

4.12 Disposable VOA vials for purging

4.13 Stainless Steel spatula

5.0 REAGENTS AND STANDARDS

5.1 The inorganic reagents used should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where available. Any other reagents must demonstrate high purity without lessening the accuracy of the determination.

5.2 Organic free reagent water - All references to water in this method will be assumed to be organic free reagent water. Type I reagent water from the laboratory (See AML SOP on reagent water) is used for this analysis.

5.3 Methanol – Purge-and-trap quality demonstrated to be free of target analytes.

5.4 Hydrochloric acid (1:1 v/v), HCl.

5.5 Stock Standard Solutions – Gasoline stock standard solution and volatile petroleum hydrocarbons (VPH) surrogate stock standard solution are ordered through Ultra Scientific and are listed as certified solutions. This stock standard, which is used for initial calibration is also used for spiking matrix spike (MS), MS duplicate (MSD), laboratory control sample (LCS), and LCS duplicate (LCSD) samples. The following table represents the solutions:

CATALOG NAME	ULTRA CATALOG NUMBER	CONCENT-RATION
Unleaded Gasoline Std	RGO-601	5000-ug/mL
VPH Surrogate Std	STS-550	5000-ug/mL

5.6 Second Source Standards - These gasoline stock solutions are purchased either from Accustandard (catalog number GA-001-40X) or Supelco (catalog number 47516-0) at a concentration of 20-mg/mL.

5.7 **NOTE:** Once the stock vials have been opened, they are to be stored in 2-mL GC vials sealed with a Teflon-lined screw-cap equipped with a Minert valve. They are kept at -10° C away from any form of light. The stock standards will expire one year from the date of preparation or on the date shown by the manufacturer. Document standard solution preparation in standard logbooks.

5.8 Fresh standards should be prepared if comparison with check standards indicates a problem. It is mandatory to replace all standards after six months. A deviation of more than 20% difference from the check standard indicates a problem.

5.9 Surrogate spiking solution - The surrogate for this method is 2, 5-Dibromotoluene. To 19-mL of methanol, add 1-mL of the stock surrogate solution (5000-ug/mL). The final concentration of the surrogate spiking solution will be 250-ug/mL. This intermediate standard will expire in three months from the date of preparation.

5.9.1 Spiking 1-uL of the surrogate into 5-mLs of the aqueous sample should be equivalent to 50-ug/L in the sample for 5-mL purge. The surrogate concentration will be 10-ug/L for 25-mL purge.

5.9.2 Spiking 1-ul of the surrogate spiking solution into 5-g of soil will equal 50-ug/kg. For medium level analysis (methanol extracts), the default surrogate concentration is 100 times higher, when 50-uL of the 5000-uL methanol extract is used for analysis (default).

5.9.3 The instrument performs all surrogate spike addition operations.

5.10 Initial Calibration (ICAL) Standards - Calibration GRO standards are prepared using the stock standard from Ultra (Section 5.5). A five-point calibration curve is analyzed for soil and/or water matrices.

5.10.1 Water ICAL standards for 5-mL purge - The five-point calibration curve for water contain gasoline at 1-mg/L, 2-mg/L, 5-mg/L, 10-mg/L, and 15-mg/L. Prepare 1-mg/L water standard as shown below: In a 50-ml volumetric flask, add 10-uL of the stock gasoline standard solution (5000-ug/mL) using a pre-calibrated syringe to 50-mL of reagent water. This will produce a final concentration of 1.0-mg/L. Invert the flask 3 times and pour the contents into a 40-mls VOA vial. Seal the VOA vial with zero headspace. Prepare other concentrations using appropriate amounts as shown in Table 1.

5.10.2 Water ICAL standards for 25-mL purge - The five-point calibration curve for water contain gasoline at 0.2-mg/L, 0.4-mg/L, 1-mg/L, 2-mg/L, and 3-mg/L. Prepare 0.2-mg/L water standard as shown below: In a 50-ml volumetric flask, add 2-uL of the stock gasoline standard solution (5000-ug/mL) using a pre-calibrated syringe to 50-mL of reagent water. This will produce a final concentration of 0.2-mg/L. Invert the flask 3 times and pour the contents into a 40-mls VOA vial. Seal the VOA vial with

zero headspace. Prepare other concentrations using appropriate amounts as shown in Table 1.

5.10.3 Soil ICAL standards Low Level - The five-point calibration curve for soil contain gasoline at 1-mg/kg, 2-mg/kg, 5-mg/kg, 10-mg/kg, and 15-mg/kg. Prepare 1-mg/kg water standard as shown below: In a 40-ml VOA vial containing 5-g of clean laboratory sand add 5-mL of Type I reagent water and 1-uL of the stock gasoline standard solution (5000-ug/mL) using a pre-calibrated syringe. This will produce a final soil concentration of 1.0-mg/kg. Seal the VOA vial immediately. Prepare other concentrations using appropriate amounts as shown in Table 1.

5.10.4 Soil ICAL standards for Medium Level - The five-point calibration curve for soil contains gasoline at 100-mg/kg, 200-mg/kg, 500-mg/kg, 1000-mg/kg, and 1500-mg/kg. Prepare 100-mg/kg water standard as shown below: In a 40-ml VOA vial containing 5-mL of Type I reagent water, add 50-uL of methanol and 1-uL of the stock gasoline standard solution (5000-ug/mL) using a pre-calibrated syringe. This will produce a final soil concentration of 100-mg/kg for the analysis of 50-uL methanol extract (5-g of soil in 5-mL methanol). Seal the VOA vial immediately. Prepare other concentrations using appropriate amounts as shown in Table 1.

5.11 Initial Calibration Verification (ICV) Standards - Prepare ICV at a concentration close to the mid-calibration standard for the ICAL using a second-source standard (Section 5.6).

5.11.1 Water ICV - Prepare water ICV samples by spiking 10-uL of the 20-mg/ml second source standard into 50-mL of water for a final concentration of 4.0-mg/L. Invert the flask 3 times and pour the contents into a 40-mL VOA vial. Seal the VOA vial with zero headspace. Use this standard for 5-mL purge. Prepare ICV for 25-mL purge at 0.8-mg/L GRO concentration using 2-uL of the 20-mg/L second source standard.

5.11.2 Soil ICV - Prepare low level soil ICV samples by spiking 1-uL of the 20-mg/mL standard into 5-mL of water and 5-g of clean laboratory sand contained in a 40-mL VOA vial. This will produce a final soil concentration of 4-mg/kg. Seal the VOA vial immediately. Add 50-uL of methanol to the vial instead of the 5-g laboratory sand to prepare ICV for medium level analysis.

5.12 Laboratory Control Sample (LCS) Standards - Prepare LCS and/or LCSD at a concentration close to the mid-calibration standard for the ICAL using the same source standard as the ICAL standard (section 5.5).

5.12.1 Water LCS - Prepare 2-mg/L water standard for the 5-mL purge analysis as shown below: In a 100-ml volumetric flask, add 40-uL of the stock gasoline standard solution (5000-ug/mL) using a pre-calibrated syringe to 100-mL of reagent water. This will produce a final concentration of 1.0-mg/L. Invert the flask 3 times and pour the contents into one or two (for LCSD) 40-mls VOA vials. Seal the VOA vial(s) with zero headspace. Use one fifth of the stock standard (8-uL instead of 40-uL) to prepare LCS for 25-mL purge.

5.12.2 Soil LCS (low level) - Prepare 2-mg/kg soil standard as shown below: In a 40-ml VOA vial containing 5-g of clean laboratory sand add 5-mL of Type I reagent water and 2-uL of the stock gasoline standard solution (5000-ug/mL) using a pre-calibrated syringe. This will produce a final soil concentration of 2.0-mg/kg. Seal the VOA vial immediately. Prepare a LCS duplicate (LCSD) in the same manner.

5.12.3 Soil LCS (medium level) - Prepare medium level soil LCS/LCSD samples by fortifying 5-ml of organic free water contained in two 40-ml VOA vials with 50-uL of purge and trap grade methanol and 2-uL of the stock gasoline standard solution (5000-ug/mL). The VOA vials are capped immediately and loaded into Preceptor-II for analysis using the soil method. The final concentration of GRO for a presumed soil extract prepared from a 5-ml methanol extract of a 5-g soil sample with zero % moisture will be 200-mg/kg.

5.13 Matrix Spike (MS) Samples - Prepare MS and MSD spiking actual environmental samples at a concentration close to the mid-calibration standard for the ICAL using the same source standard as the ICAL standard (section 5.5).

5.13.1 Water MS and MSD - Preparation of MS and MSD samples will require samples contained in three 40-mL VOA vials. Open three sample vials designated for MS and/or MSD analyses (one at a time), pour the contents into a 100-ml volumetric flask, and adjust volume to the mark. For 5-mL purge, add 40-uL of the stock gasoline standard solution (5000-ug/mL) using a pre-calibrated syringe for a sample spike amount of 2-mg/L. Pour the contents of the volumetric into two 40-ml VOA vials and cap them, ensuring zero headspace. Use one of them for MS and the other for MSD. Label the MS/MSD samples appropriately. Use 8-uL of the stock standard solution to prepare MS/MSD samples with a spike amount of 0.4-mg/L for 25-mL purge.

5.13.2 Soil MS and MSD - Use the following procedures for the preparation of MS and MSD samples collected using Method 5035. Use the alternate 5030 method only for client projects in which 5035 sample collection procedures were not used.

5.13.2.1 The MS/MSD soil samples for low level analysis collected using Method 5035 are prepared by manually injecting 2-uL of the stock gasoline standard solution (5000-ug/mL) using a pre-calibrated syringe by puncturing the septum with a small-gauge needle. The final concentration of the GRO for a 5-g soil sample (default) with zero % moisture will be 2-mg/kg.

5.13.2.2 The MS/MSD soil samples for low level analysis collected using Method 5030 are prepared by manually weighing 5-g aliquots of the sample into two 40-ml VOA vials and fortifying them with 5-ml of organic-free Type I water. Add 2-uL of the stock gasoline standard solution (5000-ug/mL) to each vial. The VOA vials are capped immediately and loaded into Preceptor-II for analysis. The final

concentration of the GRO for a 5-g soil sample with zero % moisture will be 2-mg/kg.

5.13.2.3 The MS/MSD soil samples for medium level analysis (5035 and 5030A) are prepared by fortifying 5-ml of organic-free water in two 40-ml VOA vials with 50-uL of the methanol sample extract and 2-uL of the stock gasoline standard solution (5000-ug/mL). The VOA vials are capped immediately and loaded into Preceptor-II for analysis using the soil method. The final concentration of the GRO for a sample extract prepared using 10-mL methanol and 5-g soil sample with zero % moisture will be 400-mg/kg. Use smaller volumes of methanol extract if VOC concentrations are expected to exceed the upper limit of calibration. Use a maximum of 250-uL of methanol extract.

5.14 Method Blanks - Fill a 40-mL VOA vial with organic free Type I reagent water from the laboratory. Seal the VOA vial(s) with zero headspace. Prepare MB fresh everyday.

5.15 Trip Blanks - Prepare trip blanks as described for the MB. Provide two to three 40-mL vials for each trip blank sample that needs to be collected.

6.0 SAMPLE PRESERVATION HANDLING & STORAGE

6.1 Standard 40-ml glass screw-cap VOA vials with Teflon lined silicone septa may be used for collecting aqueous samples in accordance with EPA SW-846 method 5030B/5030C. Order pre-cleaned VOA vials containing the hydrochloric acid preservative directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 2 to 3 vials for each water sample. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in separate coolers designated for storing VOC samples.

6.2 Special 40-ml VOA vials for purge-and-trap of solid samples are described in the AML SOPs based on Method 5035A. The EPA SW-846 Method 5035A has several options for soil or waste sample collection. Upon receipt and log-in, samples collected on Encore cartridges, unpreserved 40-mL VOA vials, or similar devices require preservation with sodium bisulfate or methanol as described in detail under other AML SOPs specific to these procedures. Samples should be stored in separate coolers designated for storing VOC samples.

6.3 Soil samples collected with little headspace in 2-Oz or 4-Oz jars in accordance with the old 5030A (July 1992) procedure do not require preservation. They should be stored in separate coolers designated for VOC samples.

6.4 VOA vials for headspace analysis of solid samples are described in SW-846 Method 5021. Standard 125 ml wide mouth glass containers may be used for SW-846 Methods 5031 and 5032. However, the sampling procedures

described in Method 5035A may minimize analyte loss during sample preparation better than the procedures described in Methods 5031 and 5032. No AML SOPs based on 5031 and 5032 are currently available due to lack of commercial demand for these procedures. The original EPA methods should be used for implementation as the need occurs. These samples should be stored in separate coolers designated for VOC samples.

6.5 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Analytical data generated outside of the specified holding times must be considered to be minimum values only.

7 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control contain a number of regulated compounds at high concentrations. The toxicity of these compounds present in such complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of gasoline vapors, VOCs and other organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Sample vials, which are known to contain high concentrations of vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to organic vapors.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample methanol extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Aqueous and soil samples that have undergone the purge and trap procedures should be free of volatile contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters.

9.0 PROCEDURE

9.1 Chromatographic conditions: Set up gas chromatographic conditions as described for VOCs analysis (See Appendix 1 for more details) in other AML SOPs.

9.2 Purge-and-trap for initial calibration (ICAL) standards: Prepare ICAL standards as described in Section 5.10. Place the vials onto the Archon

position and analyze by FID. The Archon adds the surrogate automatically upon purging. Produce the rest of the curve according to TABLE 1. Carryout the purge-and-trap analysis procedure using the specific conditions given in the AML SOPs based on EPA SW-846 Methods 5035A and 8260B (See also Appendix 1 for details). Analysis should be documented in quantitation reports, chromatograms, peak area determination for the GRO window, etc. (see Appendix 2 for details). Calculate response factors (or calibration factors) or use regression method using an Excel spreadsheet (see Appendix 3 for details). Verify ICAL using second-source initial calibration solutions (ICV), which are prepared as described in Section 5.11. Calculate %D using the Excel spreadsheet (see Appendix 3 for details).

9.3 Carryout the purge-and-trap analysis procedure using the same conditions for soil and 5030 for water samples as those used for the ICAL.

9.3.1 Analysis of field samples (water) - Place the sample bottle, which has allowed to come to ambient room temperature, into the correct position of the Archon. The Archon will automatically add the surrogate to each sample analysis. Analyze samples using procedures that were employed for the analysis of the initial calibration standards. The process of taking a sample aliquot destroys the validity of the sample for future analysis.

9.3.2 Low Level Analysis of field soil samples (5035A) - Refer to AML SOP based on SW-846 Method 5035A for more details. For low level analysis, use one of the two sample vials preserved either by freezing or with sodium bisulfate solution. Place the sample bottle, which has been allowed to come to ambient room temperature, into the correct position of the Archon. The Archon will automatically add the surrogate to each sample requiring analysis. Analyze the sample similarly to the initial calibration. The sample is consumed during the analysis. The analyst should use the second vial for reanalysis if QC problems were encountered and the sample contained GRO below the upper calibration limit. Use a third vial (preserved with methanol or by freezing) to perform medium level analysis, if the low-level analysis indicated the presence of VOCs in excess of the upper calibration limit. Methanol preservation method should be employed for the third analysis, if additional sample containers are unavailable for a third low level analysis.

9.3.3 Medium Level Analysis of field soil samples (5035A) - Refer to AML SOP based on SW-846 Method 5035A for more details. For medium level analysis, use the sample vial that has been preserved with methanol. If freezing is employed for sample preservation, prepare a methanol extract as described in the referenced SOP. To 5-ml of water contained in a 40-mL VOA vial, add 50-uL of the methanol extract of the sample. The dilution factor is 100 when 50-uL of the 5-mL methanol extract of a 5-g sample is used for analysis. Place the vial into the correct position of the Archon.

The Archon will automatically add the surrogate to each sample analysis. Analyze the sample similarly to the initial calibration. The entire sample prepared as described above is used by the instrument during the analysis. The methanol extract is available for repeating analysis, if required.

9.3.4 Analysis of field soil samples (5030A) - This is an obsolete procedure to be used if the client fails to collect samples in accordance with the 5035A method. Open the sample bottle, which has allowed to come to ambient room temperature, and carefully weigh out approximately 5-g into a pre-weighed 40-mL VOA vial, re-weigh the vial and record the weight. Add 5-ml of water and seal. The dilution factor is 100. The Archon will automatically add the surrogate to each sample analysis. Analyze the sample similarly to the initial calibration. The entire sample prepared as described above is used by the instrument during the analysis. Use another soil aliquot for re-analysis, if required.

9.3.5 Analysis of field samples (waste) - Open the sample bottle, which has allowed to come to ambient room temperature, and carefully weigh out approximately 4-g into a pre-weighed VOA vial, re-weigh the vial and record the weight. Add 10-mL of methanol and sonicate for 2-minutes. To 5-mL of water, add 50-uL of the methanol-extracted sample. The dilution factor is 250. Place the water into the correct position of the Archon. The Archon will automatically add the surrogate to each sample analysis. Analyze the sample similarly to the initial calibration. The entire sample prepared as described above is used by the instrument during the analysis. The methanol extract is available for repeating analysis, if required.

9.3.6 If the initial analysis of a sample target analyte exceeds the initial calibration range, the sample must be re-analyzed at a higher dilution. If the blank is not free from interference, the system must be decontaminated. Sample analysis cannot continue until after the blank analysis demonstrates the instrument to be free from interference.

9.4 Pattern recognition - Gasoline is identified by GC pattern recognition by comparing the observed chromatogram with a standard chromatogram (see Appendix 2 for details).

9.4.1 GC pattern recognition should be restricted to experienced analysts who have worked with TPH compounds.

9.5 The retention time range for GROs is defined during initial calibration. The window to be used will depend on the program supported.

9.5.1 The default range is based on SW-846 method 8015B. Two specific gasoline components 2-methylpentane (F2) and 1,2,4-trimethylbenzene (L2) are used to establish the range (See Figure in Appendix 1 for details).

9.5.2 State TPH methods that may have precedence over EPA methods may have RT ranges that are different: Two gasoline components, pentane (F1) and naphthalene (L1) are used (See Figure in Appendix 1 for details).

9.5.3 Professional judgment of the analyst should be exercised to choose a window that is appropriate for the project and the samples.

9.5.4 The chromatographic area is generated by projecting a horizontal baseline between the retention times of the first and the last eluting compounds and integrating over this window (See Figure in Appendix 1 for details).

9.5.5 Tabulate the area response of the compound by integrating over the entire ion chromatogram. The beginning and ending retention times should be the same for standards and samples.

9.6 Calculate the calibration factor (CF) for the compound using the following formula:

$$CF = \frac{\text{Total area}}{\text{Concentration (mg/L)}}.$$

9.7 Calculate the average calibration factor (CF_m) and standard deviation (SD) using the five points generated.

9.8 Calculate the % RSD of the five calibration factors using the following formula for each analyte:

$$\%\ RSD = \frac{SD * 100}{CF_m}$$

The calculated % RSD should not exceed more than 20%. If the % RSD is greater than 20%, corrective action should be implemented. Corrective action would include cleaning the injection port, replacing liners and/or septum, making new standards or baking out the columns.

9.9 ICAL Options - If the % RSD of any detected compound is greater then 20%, use least square regression methods to calculate sample concentrations. For regression methods, the instrument response is treated as the dependent variable (y) and the standard amount is the independent variable (x). When "Cs" is used as "x", use "As*Cis/Ais" as "y". When "Cs/Cis" is used as "x", use "As/Ais" as "y". Do not include origin (0,0) as an extra calibration point.

9.9.1 Weighted least squares method is preferred for calculating concentrations more accurately at lower levels. A linear least squares equation (y = a*x + b) is preferred over non-linear calibration equations (y = a*x2 + b*x + c). The coefficient of determination (COD), coefficient of variation (r2) or correlation coefficient (r) should be used to measure the goodness of fit. For

regression models to be used for quantitative purposes, the COD, r2 or r must be greater than or equal to 0.99. A value of 1.00 indicates a perfect fit. Corrective action may be required if the criteria for %RSD, r, r2, or COD are not met.

9.9.2 An initial calibration (ICAL) should be considered a single event process. Consult the QA office or check applicable EPA methods (e.g. SW-846 Method 8000C and 8081A) for additional guidance on appropriate corrective action.

9.9.3 Re-fitting of ICAL – The selected model for ICAL may be subjected to an additional re-fitting check to establish representativeness of the model for each compound in each ICAL standard. This is an optional procedure, which is recommended but not required since automated data processing procedures may not be able to accommodate it. Calculate the %D between the calculated amount (Cc) for each compound in each ICAL standard from the ICAL model and its expected value (Ce). Ideally, the %D ((Cc-Ce) * 100 /Ce) for each compound in ICAL standard should meet the QC criteria for %RSD. It is possible for one or more ICAL standards to have %D exceeding the %RSD criteria, while the %RSD for ICAL model is within QC limits. The re-fitting procedure may be used to identify ICAL standards that need re-analysis or dropped from the calculation of ICAL model when re-analysis was not performed.

9.9.4 Check and optimize instrument operating conditions including automated peak integration procedures for appropriateness in addressing the chromatographic needs of the specific compound with problems.

9.9.5 If the problem appears to be associated with just one of the ICAL standards, that one standard may be reanalyzed once immediately (must be within 8-hours) after the ICAL. Discard all the results from the original analysis of the standard in question and replace it with the results from re-analysis.

9.9.6 When additional calibration standards (six or seven instead of five) are analyzed, narrowing the range of concentrations to be used for determining the goodness of fit is an acceptable procedure. Five or more standards should remain for the calculation of RSD or linear regression for each compound after one or more points are removed from the ICAL. More points (6 for second order and 7 for third order) may be needed for non-linear regression. It is generally prohibited to remove data points from within a calibration range, while still retaining the extreme ends of the calibration range. Do not discard a middle point unless it appears to have an obvious problem and six or more standards were analyzed for ICAL and re-analysis of the affected standard was not performed within the required time limits. Changes to the upper end of the calibration range will affect the need to dilute samples

above the range. A change to the lower range is not recommended since it will affect the method quantitation limit (MQL) used to report data. The default MQLs, which are based on the lowest ICAL concentration are stored in the LIMS and project-specific changes are not practical.

9.9.7 The refitting procedures may lead to failed %Ds for upper level calibration standards for average response factor methods. The analyst may take alternate corrective actions such as the analysis of sample extracts under dilution for the affected compounds.

9.9.8 The refitting procedures may lead to failed %Ds for lower level calibration standards for regression methods. The analyst may take appropriate corrective action such as flagging the sample result as an estimated amount ("J" flag) at the lower level encountered in the sample.

9.10 The term "calibration verification" (CALV) is used to describe initial verification (ICV) of the initial calibration (ICAL) using second-source standards and subsequent continuing verifications (CCVs) using same-source standards. The response factors calculated during CALV are not used for sample quantitation since CALV standards are not used as "continuing calibration" standards permitted in some of the EPA methods.

9.11 The initial calibration is verified immediately after ICAL using a second-source ICV and it is also verified subsequently every 10 instrument injections using the same source standard (CCV). If the % drift is less than 15%, the initial calibration is assumed to be valid. If the criterion is not met (i.e., >15%), corrective action must be taken. Since the FID is quite stable, corrective action is usually associated with the injection port. If the source of the problem cannot be detected then a new five point calibration must be generated. This criterion must be met before any quantitative analysis can begin.

9.12 The calculation for the % Difference (%D) between the initial calibration (CF_m) and the calibration check (CF_v) is shown below:

$$\%D = \frac{(CF_v - CF_m) * 100}{CF_m}$$

where:

CF_m = Mean Calibration Factor from initial calibration
CF_v = Calibration factor for ICV or CCV

9.13 Analysis of field samples (water 5-mL or 25-mL purge) – The sample vials are allowed to come to room temperature. The vials are placed in the appropriate vial position. The Archon is programmed to analyze the samples according to the sequence. The surrogate is automatically added just prior to purging. Analyze the sample similarly to the initial calibration. The process of taking a volatile aliquot destroys the validity of the sample for future

analysis. The analyst should prepare a second aliquot for analysis if there is only one VOA vial. This second aliquot is maintained until the successful analysis of the first aliquot is confirmed.

9.14 Low Level Analysis of field soil samples (SW-846 Method 5030A) - This method is used when the client fails to collect samples using the 5035A method and requests analysis in accordance with State methods that do not specify sample collection using SW-846 Method 5035A. Open the sample bottle, which has allowed to come to ambient room temperature, and carefully weigh out approximately 5 grams into a VOA vial and add 5 mls of reagent water. Place the VOA vial onto the correct position. 1 ul of the surrogate is automatically added to the sample by the Archon. Analyze the sample similarly to the initial calibration.

9.15 Low Level Analysis of field soil samples (5035A) - For low level analysis, use one of the two sample vials preserved with sodium bisulfate solution. Place the sample bottle, which has been allowed to come to ambient room temperature, into the correct position of the Archon. The Archon will automatically add the ISS to each sample analysis. Analyze the sample similarly to the initial calibration. The sample is consumed during this analysis. The analyst should use the second vial for reanalysis if QC problems were encountered and the sample contained VOCs below the upper calibration limit. Use the third vial preserved with methanol to perform medium level analysis, if the low-level analysis indicated the presence of VOCs in excess of the upper calibration limit.

9.16 Medium Level Analysis of field soil samples (5035A) - For medium level analysis, use the sample vial that has been preserved with methanol. To 5 mls of water contained in 40-mL VOA vial, add 50 uLs of the methanol extract of the sample. Place the vial into the correct position of the Archon. The Archon will automatically add the ISS to each sample analysis. Analyze the sample similarly to the initial calibration. The entire sample is used by the instrument during the analysis

9.17 Analysis of field samples (waste) - Open the sample bottle, which has allowed to come to ambient room temperature, and carefully weigh out approximately 4 grams into a 40 mls VOA Vial. Add 10.00 mLs of methanol and sonicate for 2 minutes. This will serve as an intermediate dilution and extract dilution of 2.5. To 5 mLs of water, add 50 uLs of the methanol extracted sample. The dilution factor is 250. Place the 250 multiplied extract into the sample introduction inlet. Analyze the sample similarly to the initial calibration.

9.18 If the initial analysis of a sample target analyte exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. When a sample is analyzed that has saturated from compounds, this analysis must be followed by a blank water analysis. If the blank is not free from interferences, the system must be decontaminated. Sample analysis cannot continue until after the blank analysis demonstrates to be free from interference.

9.19 Perform one method blank (MB) analysis on every batch of 20 samples or less. Use procedures described in Section 5.14 for the preparation of MB.

Analyze any trip blanks (Section 5.15) that may have been collected for the project along with associated project samples.

9.20 Perform matrix spike and matrix spike duplicate analysis on every batch of 20 samples or less. Use procedures described in Section 5.13 for the preparation of MS and MSD standards.

9.21 Laboratory Control Standard (LCS) should be performed with every batch of 20 samples or less. Use procedures described in Section 5.12 for the preparation of LCS and/or LCSD standards.

10.0 SURROGATE RECOVERY CALCULATIONS

10.1 The surrogate results are calculated using the External standard method. By spiking 1 ul of the 250 ug/ml std., the concentration is 50 ug/L or 50 ug/kg for soil.

10.2 Calculate the calibration factor (CF_s) for the surrogate using the following formula:

$$CF_s = \frac{\text{Total area}}{\text{Concentration (mg/L)}}.$$

10.3 Once the surrogate standard has been quantitated and calibrated, the surrogate can be quantitatively calculated using the external standard method. After sample analysis, calculate the result of the surrogate using the following formula:

$$\text{Concentration} = \frac{\text{Total area}}{CF_s}$$

10.4 The surrogate sample result divided by the surrogate true value multiplied by 100, indicates the surrogate recovery.

11.0 CALCULATIONS

11.1 Once the samples have been purged and surrogate recoveries are acceptable, the samples results can be quantified using the Excel spreadsheet that has been used to determine calibration factors (see Appendix 3 for details).

11.2 Water samples. The following formula is used to calculate any detected analytes in a water sample:

$$\text{Concentration (mg/L)} = \frac{A_s * D}{CF_m * V_s}$$

Where:

A_s = Adjusted GC Area for the Sample
CF_m = Mean ICAL CF for the Hydrocarbon (L/mg)

D	= Dilution Factor for instrumental analysis
V_s	= Sample Volume (L)

11.3 Soil Samples

$$\text{Concentration (mg/Kg)} = \frac{A_s * D * 100}{CF_m * W_s * (\%S)}$$

Where:

A_s	= Adjusted GC Area for sample
CF_m	= Mean ICAL calibration factor (kg/mg)
D	= Dilution Factor for instrumental analysis
W_s	= Sample Wet Weight (Kg)
%S	= % Solids

12.0 QUALITY CONTROL

12.1 Analytical data, which includes calibration data, sample preparation logs, sample data and quality control data should be gathered prior to review. Checklists (see Appendix 4 for details) should be used to document raw data packages and for the verification of calculations and adherence to method procedures. Exceptions should be documented. Samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on these checklists. Summarize sample results as TPH-Gasoline or GRO on spreadsheets (see Appendix 3 for details). Summarize QC results (MB, MS/MSD, and LCS/LCSD) in the same spreadsheet.

12.2 Surrogate recovery should be within QC limits for samples, spikes and blanks. The QC limits are 80-120% for water and 75-125% for soil. Flag recoveries that are outside the QC limits with an asterisk (*). If re-extraction and/or reanalysis has been performed, and the surrogate recoveries for reanalysis are within QC limits, report data based on acceptable surrogate recoveries. If the surrogate recoveries are outside the QC limits for the reanalysis, report data for both analyses with surrogate recovery flags.

12.3 A method blank (MB) must be performed on every batch of samples, every 20 samples, or every 12 hours whichever is appropriate. Report method blank or QC association in Form-4 (see Appendix 7 for details). Include run logs and sample extraction sheets (when applicable) in project folders. Ideally, the MB should be free of target compounds. The MB level should never exceed project action levels. Report GRO in the MB at levels greater than the reporting limit (RL) using J qualifiers if the levels are less than PQL. The default action level for method blank is one half of the MQL. See Tables 2 and 3 for a list of MDLs, and MQLs. Samples associated with the method blank containing TPH-Gasoline at levels greater than one half the MQL will

require re-extraction and reanalysis unless other criteria acceptable to the client are used.

12.4 LCS percent recoveries (%R) should be within laboratory quality control (QC) limits (70 to 130%) for soil and water. Flag recoveries that are outside the QC limits with an asterisk (*). Contact supervisor for appropriate corrective action if GRO recoveries are outside the QC limits. Data qualification, re-extraction, and/or reanalysis of associated samples may be necessary depending on project requirements, turnaround time and other constraints. Document LCS/LSD results in Form-1 equivalents and recoveries/RPD in Form-3 equivalents (see Appendix 5 for details).

12.5MS recoveries should be within QC limits for the target compounds. Flag recoveries that are outside the QC limits (60 to 140% for soil and water) with an asterisk (*). Contact supervisor for appropriate corrective action if GRO recoveries are outside the QC limits. Data qualification, re-extraction, and/or reanalysis of associated samples may be necessary depending on project requirements, turnaround time and other constraints. Document MS/MSD results in Form-1 equivalents and recoveries/RPD in Form-3 equivalents (see Appendix 6 for details)

12.6Duplicates - The RPD for duplicates (sample or MS duplicates) should be within QC limits (<30%).

12.7Report GRO that is present at levels greater than the MDL (see Appendix 9 for details) or the reporting limit (RL) as appropriate using "J" qualifiers if the levels are less than MQL. GRO detected in the associated MB should also be qualified with a "B" code. If the concentration of GRO exceeds the upper limit of calibration (ICAL), flag the result with an estimated or "E" flag. The extracts may need to be diluted and reanalyzed until the concentrations are within the calibration limits for normal turnaround analyses. For rapid turnaround analyses sample results may be reported with "E" qualifiers if sufficient time is not available for reanalysis. Report sample results in Form-1 equivalents (see Appendix 10 for details).

13.0 DATA VERIFICATION AND VALIDATION - This section will provide guidance and procedures to assess if data produced using this SOP is valid and legally defensible. Document review and comments on the checklists (See Appendix 4 for details). Verify that sample preparation logs are complete and the instrument run logs are available for review (See Appendix 7 for details). Anomalies and exceptions should be documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on the checklists. Army (USACE) and Air Force (AFCEE) projects may have special requirements (See Appendix 11) that will require extra attention. Requirements may also be from the proposed implementation of the Department of Defense Quality Systems Manual (QSM) for Environmental Laboratories (Final Revision 2, June 2002), that will govern analyses performed for Army, Navy, Air Force, and the Marine Corps. A simple, universal procedure for evaluating and choosing appropriate corrective action doe not

exist to deal with situations when one or more QC parameters are outside the acceptance window. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints.

13.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) or air force (AFCEE) projects may have additional requirements that are related to USACE laboratory validation (CX), USACE District requirements, government contract requirements, specific project requirements or Architect/Engineer (A/E) requirements. Contact supervisor for specific client requirements (see Appendix 11 for details).

13.1.1 The USACE and/or AFCEE requirements and guidance from DoD QSM (see Appendix 11 for details) and elsewhere in this SOP are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may or may not be attainable for routine sample analysis. Since MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

13.1.2 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality objectives and/or other QC requirements. Exceptions and data qualifications necessary should be documented on the checklists and eventually on case narratives.

13.1.3 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist, perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete.

13.1.4 For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete.

13.1.5 For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director

will review the checklists (initial/date as necessary), prepare case narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

13.2 HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

- 13.2.1 WATER - The holding time for a sample preserved with acid is 14 days from sampling.
- 13.2.2 SOIL – A 14 day holding is used for soil and waste samples.
- 13.2.3 Holding times are established by comparing the chain of custody sampling date with the date of the injection after purging.
- 13.2.4 ACTION – If holding times have been exceeded, contact the client. All positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or invalid (I).

13.3 INITIAL CALIBRATION (ICAL) – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the GC/FID is capable of producing data within a range that is linear. For USACE and AFCEE projects the ICAL is verified (ICV) using a second source standard (see Appendix 11 for details).

- 13.3.1 %RSD for GRO – Check to see that the 20% criteria is met. If it is not met, check to see if linear regression can be used. The limit for "r" used in linear regression is 0.990. Otherwise, correct the problem and repeat the initial calibration process.
- 13.3.2 ACTION – Positive results for compounds not meeting the criteria may need to be qualified as estimated (J) by the user (client). Report detects and non-detects without qualifiers and mention these outliners in the case narrative.

13.4 CALIBRATION VERIFICATION – The calibration verification consists of two parts: (a) Initial calibration verification (ICV), which is performed using a second source standard to check on the ICAL standard; and (b) Continuing calibration verification (CCV), which is used to check that the instrument is operating under the linear range of the initial calibration. The CCV is also used to check the stability of the machine every 12 hours.

- 13.4.1 Check to see if the %D (%drift or %difference) for GRO is 15% or less for ICV and CCV.
- 13.4.2 ACTION – If the %D is >15%, check the ICAL standard and the ICV standard. Replace the ICV standard with a fresh standard and repeat ICV. If the problem is not solved, replace the ICAL

standards. Replace the intermediate and stock standards, if necessary, if the problem is not solved.

13.4.3 ACTION - If the %D is >15% for CCV, correct the problem as described for ICV and repeat the calibration verification process.

13.4.4 ACTION - If the standard analyzed after a group of samples exhibits a response that is above the 15% limit (>115%) and gasoline was not detected in the samples, then those samples do not need to be re-analyzed, as the verification standard has demonstrated that gasoline would have been detected had it been in the samples. If gasoline had been detected, then it would be necessary to re-analyze the samples with an acceptable verification standard or new ICAL.

13.4.5 ACTION - If gasoline was not detected in the samples, but the verification standard was below the ICAL response by more than 15% (<85%), then re-analysis of the samples would be necessary.

13.4.6 ACTION - If CCV failures are noted, positive results for compounds not meeting the criteria may be qualified as estimated (J) by the user (client). Report non-detects without qualifiers and mention these outliers in the case narrative.

13.5 METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample handling and processing procedures, from the field to the laboratory. According to SW-846 the MB result should be less than the MDL for the analyte, less than 5% of the regulatory limit associated with an analyte, or less than 5% of the sample result for the analyte, whichever is greater. Sample data qualification based on all the blank results (laboratory MB, equipment blank, trip blank, and other field blanks) is performed by the user (client).

13.5.1 Method blanks should be free of all target analytes and other interfering non-target analytes. To be acceptable, blank results should not exceed one half of the applicable MQL.

13.5.2 Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples. If levels greater than one half the MQLs are present, inspect and correct the problem. Reanalyze all associated samples. Contact client if other criteria are applicable. The only exception may be quick turn around projects.

13.5.3 ACTION – All positive results are reported with a “B” code, if contaminants are found in the method blank.

13.5.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, then no action is taken.

13.5.5 ACTION – Any compound detected in the sample which were also detected in associated method blank at levels greater than one half the MQLs should be re-extracted unless other client criteria are applicable.

13.5.6 ACTION – If contaminants resulting in GC/FID peak saturation are present, all affected compounds are qualified as invalid (I) and the associated samples are reanalyzed.

13.6 SURROGATE RECOVERIES – Surrogates or system monitoring compounds are different structurally, but similar chemically to classes of target compounds. These surrogate compounds are added just prior to extraction of the samples and the percent recoveries of the surrogates are calculated and evaluated.

13.6.1 Verify that the surrogate recoveries are within QC limits. Flag outliers with an asterisk (*).

13.6.2 The QC limit for aqueous samples, which includes associated QC samples (MB, LCS, LCSD, MS, and MSD) is 80% to 120%.

13.6.3 The QC limit for soil, sediment and waste samples, which includes associated QC samples (MB, LCS, LCSD, MS, and MSD) is 75% to 125%.

13.6.4 ACTION – When surrogate recoveries are outside control limits the sample must be re-analyzed. Reanalysis may be performed under dilution if original analysis should indicate high levels of target and non-target analytes. If surrogate recoveries are still outside the limits, report both sets of data and describe the problem in the case narrative.

13.7 LABORATORY CONTROL STANDARD (LCS) – A LCS analysis is used to evaluate method competence in the absence of matrix effects. The LCS should use the source standard as that of ICAL. The LCS may provide a mechanism to check method efficiency for target compounds without the sample matrix as a factor.

13.7.1 The QC limits for LCS recoveries are shown in Tables 2 and 3. The observed LCS recovery should be within QC limits (70-130% for water and soil).

13.7.2 Check the results for the LCS recoveries to see if the results are within the acceptable range. Flag outliers with an asterisk (*).

13.7.3 ACTION –If significant deviations are noticed, check retention windows, integration parameters, chromatographic baseline, and calculations. Correct any observed problem. Reanalysis of the LCS and associated samples may be necessary depending on client and project requirements. Re-extraction may also be necessary if reanalysis does not solve the observed problem. Check with the project manager about such project requirements. Professional judgment may be used by the user (client) in evaluating sample data qualification options in conjunction with other QC data for the project.

13.8 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike data are used to evaluate the precision and accuracy of the analytical method on project samples.

13.8.1 The QC limits for MS and MSD recoveries (60 to 140% for soil and water) are shown in Tables 2 and 3. The QC limit for relative percent difference (RPD) for MS duplicates (<30% for soil and water) is also shown in Tables 2 and 3. Check the MS/MSD recoveries and RPDs against QC limits. Flag outliers with an asterisk (*).

13.8.2 Perform one manual calculation using raw data and extraction sheet information.

13.8.3 NOTE – MS data evaluation is more complex than method blank or LCS data evaluation since they are complicated by matrix effects in addition to sample preparation and analysis errors. The heterogeneity of grab soil samples, and potential non-representativeness of sequentially collected water samples, etc. further complicate the evaluation since it is assumed that the native concentrations are constant in triplicate analyses. In addition, concentrations of the target analytes in the sample can also far exceed spike amounts. If the native concentration of target analytes in the sample chosen for spiking is high relative to the spiking concentration, the differences in the native concentration between the unspiked sample and the spiked samples may also become very significant.

13.8.4 NOTE - If no project samples are collected for MS analysis, samples from another project used for MS analysis may not be representative of the project samples. When project samples (e.g. surface soil) are chosen, their MS recovery characteristics may be different from those of other samples (e.g. sub-surface soil) in the same batch.

13.8.5 ACTION - The results of the MS are evaluated, in conjunction with other QC information, to determine the effect of the matrix on the bias of the analysis. The supervisor should be consulted if this is not achievable. Matrix spike recoveries that fail to meet the appropriate acceptance criteria may indicate that a potential matrix effect is present. A review of the MSD result, if available, may confirm the matrix effect, if it is in the same direction and same order of magnitude. Check the LCS and/or LCSD recoveries for affected compounds to rule out laboratory problems.

13.8.6 ACTION - If the sample chosen for MS/MSD is inappropriate, notify the client. When additional payments are authorized for re-analysis, re-extract and/or reanalyze the MS/MSD samples after employing cleanup procedures (if applicable), dilution techniques to minimize matrix interference, or using other project samples that appear to be clean, when sufficient quantities are available. Re-spike the sample at a higher level (e.g., at approximately two to four times the sample concentration), then reanalyze the sample based on project-specific requirements. It is the responsibility of the client at all times to select appropriate samples for MS

analysis, make payments for additional analyses, and to offer necessary guidance as needed to meet their data quality objectives.

13.8.7 ACTION - If the matrix effect cannot be resolved, discuss the situation in the case narrative. No action may be taken by the laboratory to qualify associated sample data based on MS/MSD recovery data alone. Professional judgment may be used by the user (client) in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

13.9 TARGET COMPOUND IDENTIFICATION AND DOCUMENTATION – Criteria for petroleum product identification are to minimize the number of erroneous positive or negative identifications of petroleum products. Document sample analyses by generating instrument printouts and Excel spreadsheets (see Appendix 2 and 3 for details). Document review in the checklist (see Appendix 4 for details). Compare observed results for each sample with adjusted MDLs, MQLs and associated method blank results to determine appropriate data qualification on sample reports (Form-1 equivalents). See AML SOP on MDLs and control charting for more details. Prepare a QC Association form (Form-4 equivalent) for samples (includes MS and MSD samples) that are associated with batch QC (MB, LCS, and/or LCSD). See Table 4 for a list of appendices that contain information useful to the analysts.

13.10 TARGET COMPOUND REPORTING AND DATA QUALIFICATION - Report compounds that were detected at concentrations that are less than the MDL and/or the lowest level of reporting (LLR) as not detected with a "U" flag. Report compounds that were detected at concentrations that are greater than the MDL and/or the LLR but lower than the MQL as estimated with a "J" qualifier. Report compounds that were detected at concentrations that are greater than the upper limit of calibration (adjusted for sample weight/volume, dilutions, and %moisture) as estimated with a "E" qualifier to indicate that calibration range has been exceeded. Reanalyze samples that contain "E" flagged data under dilution to yield data without such flags. Provide both sets of results for use by the client.

14.0 REFERENCES

14.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8015B, Volatile and Semivolatile Organic Compounds by Gas Chromatography/Flame Ionization Detector (GC/FID), December 1996.

14.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8015C, Volatile and Semivolatile Organic Compounds by Gas Chromatography/Flame Ionization Detector (GC/FID), Revision 3.

14.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8015D, Nonhalogenated Organics using GC/FID, Revision 4, June 2003.

14.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.

14.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

14.6 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5000, Sample Preparation for Volatile Organic Compounds, December 1996.

14.7 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5021, Volatile Organic Compounds in soils and other solid matrices using equilibrium headspace analysis, December 1996.

14.8 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5030A, Purge and Trap, July 1992.

14.9 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5030B, Purge and Trap for Aqueous Samples, December 1996.

14.10 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5030C, Purge and Trap for Aqueous Samples, Revision 3, May 2003.

14.11 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5031, Volatile Nonpurgeable Water-soluble Compounds by Azeotropic Distillation, December 1996.

14.12 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5032, Volatile Organic Compounds by Vacuum Distillation, December 1996.

14.13 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5035, Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples, December 1996.

14.14 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5035A, Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples, Draft Revision 1, July 2002.

14.15 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5041A, Analysis for Desorption of Sorbent Cartridges from Volatile Organic Sampling Train (VOST), December 1996.

14.16 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3585, Waste Dilution for Volatile Organics, December 1996.

14.17 Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, EPA, 40 CFR Part 136, Method 624: Purgeables, October 1984.
14.18 Methods for the Determination of Organic Compounds in Drinking Water, Supplement II, EPA/600/R-92/129, Method 524.2, Measurement of Purgeable Organic Compounds in Water by Capillary column Gas Chromatography / Mass Spectrometry, August 1992.
14.19 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.
14.20 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.
14.21 AML Standard Operating Procedures, AML-5035-SA1.
14.22 AML Standard Operating Procedures, AML-5035-SC1.
14.23 AML Standard Operating Procedures, AML-5035-SP1.
14.24 AML Standard Operating Procedures, O-5035-RA.
14.25 AML Standard Operating Procedures, O-SoilVOCs-5035A-RB.
14.26 AML Standard Operating Procedures, Q-Water-RC.
14.27 AML Standard Operating Procedures, Q-WasteMgmt-RD.
14.28 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
14.29 AML Standard Operating Procedures, Q-Controlcharts-RC.
14.30 Determination of total purgeable (gasoline range) petroleum hydrocarbons (OA-1), State of Iowa, 1991.

TABLE 1

PREPARATION OF DAILY INITIAL CALIBRATION (ICAL) STANDARDS USING THE 5000-ug/mL STOCK SOLUTION

Amt. of STD (uL)	**Water 5-mL Purge** Volume (mL) Water	Final Conc (mg/L)	Amt of STD (uL)	**SOIL Low Level** Weight (grams)	Final Conc (mg/kg)
10	50	1	1	5	1
20	50	2	2	5	2
50	50	5	5	5	5
100	50	10	10	5	10
150	50	15	15	5	15

Amt. of STD (uL)	**Water 25-mL Purge** Volume (mL) Water	Final Conc. (mg/L)	Amt. of STD (uL)	**SOIL Medium Level** Weight (grams)	Final Conc. (mg/kg)
2	50	0.2	1	0.05	100
4	50	0.4	2	0.05	200
10	50	1.0	5	0.05	500
20	50	2.0	10	0.05	1000
30	50	3.0	15	0.05	1500

Table 2
Aqueous Sample MDL, MQL, and QC Limits for LCS and MS

Total Petroleum Hydrocarbons (TPH) Analysis by GC/FID

Analytical Management Laboratories

Matrix: Water	**Sample Preparation**		**Sample Analysis**			**Technique**
Parameter	Method No.		Method No.		State UST	
GRO (5-mL sample)	5030C		8015D		OA1/GRO	Purge and Trap GC/FID
GRO (25-mL sample)	5030C		8015D		OA1/GRO	Purge and Trap GC/FID

	Spike Amount	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**		**LCS Limits %R**		**MS Limits %R**		**RPD Limit**
Parameter	(mg/L)	**Mg/L B**	mg/L	**mg/L A**	RD= B/A	flag	**mg/L C**	RQ= C/A	flag	Lower	Upper	Lower	Upper	%
GRO (5-mL purge)	0.15	0.103	0.026	**0.082**	1.3		1.0	12.2		70	130	60	140	30
GRO (25-mL purge)	0.05	0.074	0.003	**0.008**	9.2		0.2	25.0		70	130	60	140	30

GRO = Gasoline Range Hydrocarbons or TPH-Gasoline (Purgeable).

OA1 is State (Iowa, Kansas, and Missouri) UST method based on SW 8015 for GRO.

Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 10 for multicomponent analytes.

Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.

LCS and MS %R limits are laboratory limits for multicomponent analytes with guidance from USACE EM200-1-3, Appendix I.

Table 3
Soil/Sediment Sample MDL, MQL, and QC Limits for LCS and MS

Total Petroleum Hydrocarbons (TPH) Analysis by GC/FID

Analytical Management Laboratories

Matrix: Soil	**Sample Preparation**		**Sample Analysis**			**Technique**
Parameter	Method No.		Method No.		State UST	
GRO	5035A		8015D		OA1	Purge and Trap GC/FID

	Spike Amount	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**		**LCS Limits %R**		**MS Limits %R**		**RPD Limit**
Parameter	(mg/kg)	**Mg/kg B**	mg/kg	**mg/kg A**	RD=B/A	flag	**mg/kg C**	RQ=C/A	flag	Lower	Upper	Lower	Upper	%
GRO	0.25	0.151	0.014	**0.043**	3.5		1.0	23.5		70	130	60	140	30

GRO = Gasoline Range Hydrocarbons or TPH-Gasoline (Purgeable).

OA1 is State (Iowa, Kansas, and Missouri) UST method based on SW 8015 for GRO.

Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 10 for multicomponent analytes.

Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.

LCS and MS %R limits are laboratory limits for multicomponent analytes with guidance from USACE EM200-1-3, Appendix I.

Table 4
List of Appendices

Appendix Number	Appendix Topic
1	Chromatogram for the Gasoline Standard Retention Time Ranges for Gasoline Range Organics
2	Quantitation Reports, Chromatograms, and peak areas for GRO Instrument Printouts
3	Initial Calibration (ICAL) Summary (Form-6 equivalent benchsheets) Calibration Verification Summary (Form-7 equivalent benchsheets) Sample Results Summary (Form-1 equivalent benchsheets) Surrogate, LCS and MS Summary (Form-2 and 3 equivalent benchsheets)
4	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
5	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
6	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
7	QC Association Forms (Form-4 equivalents) Sample Preparation Logsheets Instrument Run Log
8	Surrogate Recovery Reports (Form-2 equivalents)
9	Method Detection Limit (MDL) Study Summary
10	Sample Reports (Form-1 equivalents)
11	Client Method Quality Objectives for Method 8015 mod. (AFCEE, USACE, DoD QSM, etc.)

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. O-EDB-DBCP-TCP-RC

EDB, DBCP, and 1,2,3-TCP by Microextraction and GC/ECD Analysis (EPA Methods 504, 504.1 and SW846-8011)

October 28, 2005

APPROVED:

_____ _____
Laboratory Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

EDB, DBCP, and 1,2,3-TCP by Microextraction and GC/ECD Analysis (EPA Methods 504, 504.1 and SW846-8011)

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) based on EPA methods 504.1 and SW-846 801181A) is used to determine the concentrations of ethylene dibromide or 1,2-Dibromoethane (EDB, CAS # 106-93-4), 1,2-Dibromo-3-chloropropane (DBCP, CAS # 96-12-9), and 1,2,3-Trichloropropane (TCP, CAS# 96-18-4) in water samples.

1.2 This SOP employs microextraction and analysis by gas chromatography using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD).

1.3 The experimentally determined method detection limits (MDL) for EDB and DBCP were calculated to be 0.01 µg/L. The method has been shown to be useful for these analytes over a concentration range of approximately 0.03 to 200 µg/L. Actual detection limits are highly dependent upon the characteristics of the gas chromatographic system, sample matrix, and calibration. See Table 1 for MDLs obtained at the laboratory.

1.4 This method is restricted to use by or under the Supervision of analysts experienced in the use of gas chromatography and in the interpretation of gas chromatograms. Each analyst should demonstrate the ability to generate acceptable results with this method.

1.5 EDB, DBCP, and TCP have been tentatively classified as known or suspected human or mammalian carcinogens. Pure standard materials and stock standard solutions of these compounds should be handled in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

2.0 SUMMARY OF METHOD

2.1 Thirty five mL of sample are extracted with 2 mL of hexane. Two µL of the extract are then injected into a gas chromatograph equipped with a linearized electron capture detector for separation and analysis.

2.2 Calibration standards, laboratory control samples (LCS) and aqueous matrix spikes (MS) are extracted and analyzed in an identical manner as the samples in order to compensate for possible extraction losses.

2.3 The extraction and analysis time is 30 to 50 minutes per sample depending upon the analytical conditions chosen.

2.4 Confirmatory evidence is obtained using a different column.

3.0 INTERFERENCES

3.1 Impurities contained in the extracting solvent (hexane) usually account for the majority of the analytical problems. Reagent blanks should be analyzed for each new bottle of hexane before use. Indirect daily checks on the hexane are obtained by monitoring the reagent blanks. Whenever an interference is noted in the method or instrument blank, the laboratory should reanalyze the hexane and obtain a new source of hexane solvent. Interference-free hexane is defined as containing less than 0.01-µg/L of the analytes. Protect interference-free hexane by storing it in an area known to be free of organochlorine solvents.

3.2 Several instances of accidental sample contamination have been attributed to diffusion of volatile organics through the septum seal into the sample bottle during shipment and storage. Trip blanks must be used to monitor for this problem.

3.3 This liquid/liquid extraction technique extracts a wide boiling range of non-polar organic compounds and, in addition, extracts some polar organic compounds. EDB at low concentrations may be masked by very high concentrations of dibromochloromethane (DBCM), a common chlorinated contaminant in drinking water samples.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph - A Hewlett Packard 5890 Series II GC is used. The system is complete with a temperature-programmable GC for splitless injection. The system includes all accessories, including syringes, analytical columns and gasses. The column flow rate remains constant throughout temperature program operation. The capillary column is directly coupled to the source.

4.2 Gas chromatographic columns

4.1.1 Column 1 - This column (30 meter x 0.32 mm ID Durawax-DX 3, 0.25 µm film thickness) is recommended as the primary column in the referenced methods.

4.1.2 Column 2 - This column (30 meter x 0.32 mm ID DB-1, 0.25 µm film thickness) is recommended as the confirmation column in the referenced methods.

4.1.3 Column 3- A 30 meter x 0.32 mm ID (0.25 µm film thickness) capillary column coated with DB-5 (J&W Scientific) is employed at the laboratory for routine analysis.

4.1.4 Column 4- A 30 meter x 0.32 mm ID (0.25 µm film thickness) capillary column coated with DB-1701 (J&W Scientific) is employed at the laboratory for confirmation.

4.3 Data system - Hewlett Packard's Environquant is used to allow continuous acquisition and storage on machine-readable media of all data obtained throughout the duration of the GC program.

4.4 Microsyringes - 10, 25, 100, 250, 500 and 1000 uLs.

4.5 Balance - Analytical, 0.0001 grams and top loading, 0.1 grams.

4.6 Disposable pipettes - Pasteur

4.7 Microsyringes - 10, 25, 50, 100, 500, 1000 uLs

4.8 Volumetric flasks, Class A - 10 mL.

4.9 Glass bottles - 15 mL, with Teflon lined screw caps or crimp tops.

4.10 Graduated cylinder - 50 mL.

4.11 Transfer pipet.

5 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - Use Type 1 water (see applicable AML SOP on reagent water).

5.3 Hexane, C_6H_{14} - UV grade (Burdick and Jackson #216 or equivalent).

5.4 Methyl alcohol, CH_3OH - Demonstrated to be free of analytes.

5.5 Sodium chloride, NaCl - Pulverize a batch of NaCl and place it in a muffle furnace at room temperature. Increase the temperature to 400°C for 30 minutes. Store in a capped bottle.

5.6 1,2-Dibromoethane (99%), $C_2H_4Br_2$, (Aldrich Chemical Company, or equivalent).

5.7 1,2-Dibromoe-3-chloropropane (99.4%), $C_3H_5Br_2C1$, (AMVAC Chemical Corporation, Los Angeles, California, or equivalent).

5.8 1,2,3-Trichloropropane (99%), Aldrich Chemical Company or equivalent.

5.9 EPA 504.1 mix (Supelco)

5.10 Stock standards - These solutions may be purchased as certified solutions or prepared from pure standards using the following procedures:

5.10.1 In-house Preparation - Place about 9.8 mL of methanol into a 10 mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes and weigh to the nearest 0.0001 g. Use a 25 µL syringe and immediately add two or more drops (10-µL) of standard to the flask. Be sure that the standard falls directly into the alcohol without contacting the neck of the flask. Reweigh, dilute to volume, stopper, and then mix by inverting the flask several times. Calculate the concentration in milligrams per liter (mg/L) from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard.

5.10.2 Purchase DBCP/EDB/TCP mixture (Catalogue No. HCM-812) at a concentration of 2000-µg/mL in methanol from Ultra Scientific.

5.10.3 Store stock standards in amber vials equipped with Teflon lined screw-caps or crimp tops. Stock standards are stable for at least four weeks when stored at 4°C and away from light.

5.11 Intermediate Standard - Use stock standards to prepare an intermediate standard that contains both analytes in methanol. The intermediate standard should be prepared at a concentration that can be easily diluted to prepare aqueous calibration standards that will bracket the working concentration range. Store the intermediate standard with minimal headspace and check frequently for signs of deterioration or evaporation,

especially just before preparing calibration standards. The storage time described for stock standards also applies to the intermediate standard.

5.11.1 Prepare Intermediate Standard-1 (IS-1) by diluting 100-µL of the Ultra Stock Standard (Section 5.8.2) to 10-mL in methanol. The concentration of the target analytes in IS-1 is 20-µg/mL. Store IS-1 in amber vials equipped with Teflon lined screw-caps or crimp tops. The IS-1 should be stable for at least four weeks when stored at 4°C and away from light.

5.11.2 Prepare Intermediate Standard-2 (IS-2) by diluting 100-µL of the Intermediate Standard-1 (Section 5.9.1) to 10-mL in methanol. The concentration of the target analytes in IS-2 is 0.2-µg/mL. Store IS-1 in amber vials equipped with Teflon lined screw-caps or crimp tops. The IS-1 should be stable for at least four weeks when stored at 4°C and away from light.

5.12 Initial Calibration Standards - A minimum of 5 initial calibration solutions (ICAL) are prepared. Various volumes of the IS-2 solution is diluted in 35-mL (final volume) of reagent water to prepare the ICAL standards. Measure out 35-mL of reagent water into 40-mL VOA vials. Remove volumes of water that equal the volume of IS-2 solution that will be spiked into them. No such correction is necessary for small volumes (<0.5-mL). Immediately after spiking the IS-2 solution, repace the cap and swirl the container for uniform mixing. Prepare ICAL solutions as described below:

5.12.1 ICAL-1. Prepare this solution (0.0174-µg/L) by spiking 3-µL of IS-2 into 35-mL reagent water.

5.12.2 ICAL-2. Prepare this solution (0.057-µg/L) by spiking 10-µL of IS-2 into 35-mL reagent water.

5.12.3 ICAL-3. Prepare this solution (0.143-µg/L) by spiking 25-µL of IS-2 into 35-mL reagent water.

5.12.4 ICAL-4. Prepare this solution (0.286-µg/L) by spiking 50-µL of IS-2 into 35-mL reagent water.

5.12.5 ICAL-5. Prepare this solution (0.429-µg/L) by spiking 75-µL of IS-2 into 35-mL reagent water.

5.12.6 ICAL-6. Prepare this solution (0.571-µg/L) by spiking 100-µL of IS-2 into 35-mL reagent water.

5.12.7 ICAL-7. Prepare this solution (1.142-µg/L) by spiking 200-µL of IS-2 into 35-mL reagent water.

5.12.8 ICAL-8. Prepare this solution (2.285-µg/L) by spiking 400-µL of IS-2 into 35-mL reagent water.

5.12.9 **NOTE:** Eight ICAL solutions are <u>not</u> required. A few intermediate points, the lowest or the highest point may be left out to accommodate the anticipated concentrtaion range. Samples with EDB and/or DBCP concentrations that are greater than 2-µg/L may be measured quantitatively with other VOC methods (e.g. 524.2, 8260B, etc.).

5.13 Initial Calibration verification (ICV) or Quality control reference (QC) standards - Prepare a QC reference sample concentrate at 0.286-µg/L of both analytes from

standards from a different source than the standards used for the stock standard. Prepare as described in Section 5.10.4 using a second source IS-2 standard (0.2-µg/mL).

5.14 Continuing calibration Verification Standard (CCV) - Prepare several CCV solutions at 0.286-µg/L of both analytes from standards from the same source as the ICAL standards. Prepare exactly as described (Section 5.10.4) for ICAL-4.

5.15 Method Blank - Prepare method blank (MB) using the same procedure as ICAL standards. The spike volume is zero

5.16 Laboratory Control Samples - Prepare exactly as described for CCV (Section 5.12). Prepare in duplicate (LCS and LCSD).

5.17 Matrix Spike Samples - Prepare in duplicate. Remove the container cap from two vials designated for MS and MSD analyses. Remove and discard a 5-mL volume using a 5-mL transfer pipet. Replace the container cap and weigh the container with contents to the nearest 0.1-g. Record this weight for subsequent sample volume determination. See section 9.7 for determining sample volume. Spike 50-µL of IS-2 solution into each vial. Immediately after spiking the IS-2 solution, repace the cap and swirl the container for uniform mixing. The spiked MS and MSD samples are now ready for analysis.

5.18 Surrogate - A surrogate has not been identified in any of the EPA methods (EPA 504.1 and SW-846 8011) referenced in this SOP. The Air Force (see AFCEE QAPP for details) recommends the use of 1,2-Dibromopropane (DBP) or 1,2-Dichloropropane (DCP) as surrogate. The laboratory will be using DCP as the surrogate.

5.18.1 Acquire neat standard of 1,2-Dichloropropane (CAS #78-87-5, Catalogue No. RHH-037) from Ultra Scientific.

5.18.2 Dissolve 20-mg of the pure compound in 10-mL methanol to prepare 2000-µg/L Stock Surrogate Standard (SSS). Store SSS in amber vials equipped with Teflon lined screw-caps or crimp tops. The SSS should be stable for at least one year when stored at 4°C and away from light.

5.18.3 Prepare Intermediate Surrogate Standard-1 (ISS-1) by diluting 100-µL of the Stock Surrogate Standard (Section 5.16.2) to 10-mL in methanol. The concentration of DCP in ISS-1 is 20-µg/mL. Store ISS-1 in amber vials equipped with Teflon lined screw-caps or crimp tops. The ISS-1 should be stable for at least one year when stored at 4°C and away from light.

5.18.4 Prepare Intermediate Surrogate Standard-2 (ISS-2) by diluting 1000-µL of the Intermediate Surrogate Standard-1 (Section 5.16.3) to 10-mL in methanol. The concentration of DCP in ISS-2 is 2.0-µg/mL. Store ISS-2 in amber vials equipped with Teflon lined screw-caps or crimp tops. The ISS-2 should be stable for at least one month when stored at 4°C and away from light. Use 50-µL of the ISS-2 solution to spike all calibration standards (ICAL, ICV, CCV) and samples including QC samples (MB, LCS, LCSD, MS, and MSD).

5.18.5 Spike 50-µL of the ISS-2 to each sample. The concentration of DCP in aqueous samples (35-mL) will be 2.86-µg/L. The default DCP concentration for soil samples (7-g) will be five times higher at 14.3-µg/kg.

6 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

6.1 Groundwater, Surface water, and Wastewater samples - Standard 40-ml glass screw-cap VOA vials with Teflon lined silicone septa may be used for collecting these aqueous samples. Order pre-cleaned VOA vials not containing the hydrochloric acid preservative directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 2 vials for each water sample.

6.1.1 Although the target analytes are volatile organic compounds (VOCs), the samples should not be preserved with hydrochloric acid. Adjustment of sample pH to prevent biological degradation of method analytes has not been tested. Therefore, the addition of acid preservatives to samples is not recommended.

6.1.2 A dechlorinating agent (sodium thiosulfate) should be added to chlorinated drinking water samples to arrest the formation of trihalomethanes such as dibromochloromethane (DBCM), which may interfere with the target compound analysis. Groundwater, surface water and wastewater samples that have not been chlorinated do not require the addition of sodium thiosulfate.

6.1.3 Since the target analytes for this SOP are VOCs, the analysis of trip blanks (equivalent to field reagent blanks described in Section 8.1.1 of EPA 504.1) is required. Fill two 40-mL VOA vials with Type I water without headspace and ship them to the sampling site with empty sample containers. One trip blank sample (set of two vails) should accompany the environmental samples collected in the field during shipping and storage.

6.1.4 Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. Provide two sets of 2 vials for the MS and MSD samples.

6.1.5 When sampling from a well, fill a wide-mouth bottle or beaker with sample, and carefully fill 40-mL sample bottles.

6.2 Soil samples collected with little headspace in 2-Oz or 4-Oz jars do not require chemical preservation. They should be stored in separate coolers designated for VOC samples. Soil samples may need to be accompanied by a set of trip blanks, if required by the project quality assurance plan.

6.3 Drinking water samples - The procedure described for water sampls (Section 6.1) is used with the following modifications.

6.3.1 Provide a small vial containing sodium thiosulfate preservative and a spatula per sampling event when chlorinated drinking water samples are scheduled to be collected. Adding 3-mg sodium thiosulfate per 40-mL sample should neutralize the residual chlorine that may be present in treated water. Alternately, 75-µL of a freshly prepared sodium thiosulfate solution (40-mg/mL) may be added to empty 40-mL bottles just prior to sample collection.

6.3.2 Alternatively, a set of two 40-mL vials containing 3-mg sodium thiosulfate may be provided for the collection of each chlorinated drinking water sample.

6.3.3 When sampling from a tap, open the tap and allow the system to flush until the water temperature has stabilized (usually about 10 minutes). Adjust the flow to about 500-mL/min and collect the sample from the flowing stream.

6.4 The samples must be chilled to 4°C or less at the time of collection and maintained at that temperature during shipment. Sample should be packaged with sufficient ice

during shipment for the samples to be maintained at 4°C or less on arrival at the laboratory. Upon receipt and log-in, samples should be stored in separate coolers designated for storing VOC aqueous samples at 4°C.

6.4.1 Samples must be extracted and analyzed within the specified holding times (28-days according to EPA Method 504 and 14-days if 1,2,3-trichloropropane requires analysis according to EPA Method 504.1) for the results to be considered reflective of total concentrations. Analytical data generated outside of the specified holding times should be considered to be minimum values only. Such data may be used to demonstrate that the concentration of a constituent to be above the regulatory threshold but cannot be used to demonstrate for compliance with regulatory limits.

7 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control contain a number of regulated compounds at high concentrations. The toxicity of these compounds present in such complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Aqueous and soil samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters after the solvent(s) have been allowed to evaporate in a hood.

9 PROCEDURE

9.1 Gas Chromatographic Conditions - Two gas chromatography columns are recommended in the referenced EPA methods. Column 1 is a highly efficient column that provides separations for EDB and DBCP without interferences from trihalomethanes. Column 1 can be used as the primary analytical column. Column 2 is recommended in the referenced methods for use as a confirmatory column. Column 3 is used at the laboratory as primary analytical column and column 4 is uaed for confirmation. The injected sample is split for the dual column analysis.

9.1.1 Column 1:

Injector temperature: 200°C.

Detector temperature: 290°C.
Carrier gas (Helium)Linear velocity: 25 cm/sec.
Temperature program:
Initial temperature: 40°C, hold for 4 min.
Program: 40°C to 190°C at 8°C/min.
Final temperature: 190°C, hold for 25 min.

9.1.2 Column 2:
Injector temperature: 200°C.
Detector temperature: 290°C.
Carrier gas (Helium)Linear velocity: 25 cm/sec.
Temperature program:
Initial temperature: 40°C, hold for 4 min.
Program: 40°C to 270°C at 10°C/min.
Final temperature: 270°C, hold for 10 min.

9.1.3 Column 3:
Injector temperature: 200°C.
Detector temperature: 300°C.
Carrier gas (Helium)Linear velocity: 25 cm/sec.
Temperature program:
Initial temperature: 40°C, hold for 4 min.
Program: 40°C to 270°C at 10°C/min.
Final temperature: 270°C, hold for 10 min.

9.1.4 Column 4:
Injector temperature: 200°C.
Detector temperature: 300°C.
Carrier gas (Helium)Linear velocity: 25 cm/sec.
Temperature program:
Initial temperature: 40°C, hold for 4 min.
Program: 40°C to 270°C at 10°C/min.
Final temperature: 270°C, hold for 10 min.

9.2 Calibration

9.2.1 Prepare at least five initial calibration standards (ICAL) as described in Section 5.10. The lowest ICAL standrad should contain EDB and DBCP at a concentration near, but greater than, the method detection limit (Table 1) for each compound. The others should be at concentrations that bracket the range expected in the samples.

9.2.2 Perform micro-extraction on ICAL standards as described for samples (Section 9.4) and analyze the hexane extract from each calibration standard. Tabulate peak height or area response versus the concentration in the standard. Prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (< 10% relative standard deviation), linearity can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

9.3 Aqueous Sample preparation - Prepare sample using SW-846 Method 3511 (Microextraction) with EPA 504.1/8011 modifications as described below:

9.3.1 Remove samples and standards from storage and allow them to reach room temperature.

9.3.2 Prepare calibration standards (ICAL, ICV, and CCV) as described in Section 5.10, 5.11, and 5.12.

9.3.3 Prepare batch quality control samples (MB, LCS, and LCSD) as described in Section 5.13 and 5.14.

9.3.4 Prepare MS and MSD samples as described in section 5.15.

9.3.5 Prepare other samples as described below: For samples and field blanks contained in 40-mL bottles, remove the container cap. Discard a 5-mL volume using a 5-mL transfer pipet. Replace the container cap and weigh the container with contents to the nearest 0.1-g and record this weight for subsequent sample volume determination.

9.4 Soil Sample Preparation – Prepare sample using SW-846 Method 3570 (Microextraction) with modifications as described below:

9.4.1 Mark the outside of several VOA vials with a mark at the 35-mL volume.

9.4.2 Remove the cap and weigh out 7-g of soil or sediment into the 40-mL VOA vial. Add reagent water to the mark so that the final volume of the soil and water mixture is 35-mL. Replace the cap.

9.4.3 Swirl the vial to suspend the soil/sediment in the aqueous phase. Extract the aqueous soil extract in the same manner as aqueous samples (Section 9.6 and 9.7).

9.5 Sample Extraction

9.5.1 Remove the container cap and spike 50-µL of the ISS-2 (Section 5.16.4) surrogate solution and add 7-g of NaCl to all samples and standards. Recap the sample container and dissolve the NaCl by shaking by hand for about 20 seconds. Remove the cap and using a transfer pipet, add 2.0-mL of hexane. Recap and shake vigorously by hand for 1 minute. Allow the water and hexane phases to separate. If stored at this stage, keep the container upside down.

9.5.2 Remove the cap and carefully transfer a sufficient amount (1.0-mL) of the hexane layer into a GC vial using a disposable glass pipet for GC/ECD analysis. Transfer the remaining hexane phase, being careful not to include any of the water phase, into a second vial. Reserve this second vial at $4^{o}C$ for reanalysis if necessary.

9.6 Sample Extract Analysis - Transfer the first sample vial to an autosampler set up to inject 2.0 µL portions into the gas chromatograph for analysis.

9.7 Determination of Sample Volume - Perform after removing aliquots of the hexane extract for initial analysis and reanalysis.

9.7.1 Remove the cap from the sample container. Discard the remaining sample/hexane mixture. Shake off the remaining few drops using short, brisk wrist movements. Reweigh the empty container with original cap.

9.7.2 Calculate the net weight of sample by difference between the weights recorded in Section 7.3.1 and 7.6.1 to the nearest 0.1 g.

9.7.3 This net weight is equivalent to the volume of water (V_s) extracted.

V_s (mL) = gross weight (grams) - bottle tare (grams)

9.8 Calculations

9.8.1 Identify EDB and DBCP in the sample chromatogram by comparing the retention time of the suspect peak to retention times generated by the calibration standards and the check standard.

9.8.2 Use the calibration curve or calibration factor to directly calculate the uncorrected concentration (C_i) of each analyte in the sample (e.g. calibration factor x response).

9.8.3 Calculate the sample volume (V_s) in mL as equal to the net sample weight in grams (Section 9.8.3).

9.8.4 Calculate the corrected sample concentration in aqueous and soil samples as follows:

$$\text{Concentration } (\mu g/L) = \frac{(C_i) * (V_s + 5) * DF}{V_s}$$

$$\text{Concentration } (\mu g/kg) = \frac{(C_i) * 35 * DF}{(W_s/1000) * (PS/100)}$$

where:
C_i is the concentration of the compound (µg/L) - see Section 9.9.2
V_s is the aqueous sample volume (mL) - see Section 9.8.3
W_s is the weight of the soil sample (g) - see Section 9.5
DF is the dilution factor for the hexane extract analysis
PS is the percent solids for the soil sample
35 is the default sample volume (mL) for aqueous and soil samples

9.8.5 Report the results in µg/L (µg/kg for soil based on dry weight) to three significant figures.

10.0QUALITY CONTROL

10.1Using this SOP will involve implementing the referenced EPA method at the laboratory. Method implementation involves the following: chromatographic separation of the target analytes on two GC columns, determination of retention time windows for each compound, determination of method detection limits for each sample matrix and/or preparation method, spiked sample analysis, initial demonstration of acceptable recoveries, etc. Method implementation procedures are described on the AML SOP on MDL.

10.2Analytical data, which includes calibration data, sample preparation logs, sample data and quality control data should be gathered prior to review. A review checklist

(see Appendix 5 for details) should be used to document verification of calculations and adherence to method procedures. Exceptions should be documented. Samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on these checklists.

10.3 Summarize sample results using Form-1 Equivalents (See Appendix 12). Summarize QC results and QC recoveries in Form-1 and Form-3 equivalents respectively for MS/MSD (Appendix 7) and LCS/LCSD (Appendix 8).

10.4 Surrogate recovery should be within QC limits (70 to 120% for aqueous samples and 60 to 130% for soil) samples, spikes and blanks. Document surrogate recoveries in Form-2 equivalents (See Appendix 9) Flag recoveries that are outside the QC limits with an asterisk (*). If re-extraction and/or reanalysis has been performed, and the surrogate recoveries for reanalysis are within QC limits, report data based on acceptable surrogate recoveries. If the surrogate recoveries are outside the QC limits for the reanalysis, report data for both analyses with surrogate recovery flags.

10.5 Method blanks (MB) should be free of target compounds. Report target compounds that are present in the MB at levels greater than the reporting limit (RL) using J qualifiers if the levels are less than MQL. The action level for method blank is one half of the MQL. See Tables 1 and 2 for a list of MDLs, RLs, and MQLs. Details of MDL determinations are included in Appendix 10. Samples associated with the method blank containing target compounds at levels greater than on half the MQL will require re-extraction and reanalysis unless other criteria accepable to the client are used. Since method blanks may be contaminated when they are associated with samples containing high levels of target compounds, only samples that contain less than one tenth of the MB amount may need to be re-extracted and reanalyzed.

10.6 LCS recoveries should be within QC limits for the target compounds. Flag recoveries that are outside the QC limits with an asterisk (*). See Tables 1 and 2 for QC limits. Data qualification, re-extraction, and/or reanalysis of associated samples may be necessary depending on project requirements, turnaround time and other constraints.

10.7 MS recoveries should be within QC limits for the target compounds. Flag recoveries that are outside the QC limits with an asterisk (*). See Tables 1 and 2 for QC limits. Data qualification, re-extraction, and/or reanalysis of associated samples may be necessary depending on project requirements, turnaround time and other constraints.

10.8 Report sample results on Form-1 equivalents (See Appendix 12). Report target compounds that are present at levels greater than the reporting limit (RL) using "J" qualifiers if the levels are less than MQL. Compounds that were also detected in the associated MB should also be qualified with a "B" code. If the concentration of any of the target compounds exceed the upper limit of calibration (ICAL), flag the affected compounds data with an estimated or "E" flag. The extracts may need to be diluted and reanalyzed until the concentrations are within the calibration limits for normal turnaround analyses. For rapid turnaround analyses sample results may be reported with "E" qualifiers if sufficient time is not available for reanalysis.

11.0 **DATA VERIFICATION AND VALIDATION -** This section will provide guidance and procedures to assess if data produced using this SOP is valid and legally defensible. Document review and comments on or more of the the checklists (See Appendix 5 for details). Verify that sample preparation logs are complete and the instrument run logs are available for review (See Appendix 11 for details). Anomalies and exceptions should be documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on the checklists. Army (USACE) and Air Force (AFCEE) projects may have special requirements (See Appendix 13) that will require extra attention. Requirements may also be from the proposed implementation of the Department of Defense Quality Systems Manual (QSM) for Environmental Laboratories (Final Revision 2, June 2002), that will govern analyses performed for Army, Navy, Air Force, and the Marine Corps. A simple, universal procedure for evaluating and choosing appropriate corrective action doe not exist to deal with situations when one or more QC parameters are outside the acceptance window. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints.

11.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) projects may have additional requirements that are related to USACE laboratory validation (CX), USACE District requirements, government contract requirements, specific USACE project requirements or USACE Architect/Engineer (A/E) requirements. USACE-CX requirements (Summary of Method Quality Objectives for Method 8011) are included in Appendix 13.

11.1.1 The USACE and/or AFCEE requirements shown in Appendix 13 and elsewhere in this SOP are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may or may not be attainable for routine sample analysis. Since USACE MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

11.1.2 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality objectives and/or other QC requirements. Exceptions and data

qualifications necessary should be documented on the checklists and eventually on case narratives.

11.1.3 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist, perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete. For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete. For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director will review the checklists (initial/date as necessary), prepare case narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

11.2HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

11.2.1 WATER - The holding times is 14 days from sampling.

11.2.2 SOIL – A 14 day holding is used for soil and waste samples.

11.2.3 Holding times are established by comparing the chain of custody sampling date with the date of the injection.

11.2.4 ACTION – If holding times have been exceeded, contact the client. All positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or innvalid (I).

11.2.5 NOTE – Holding time is extended to 28-days for EDB and DBCP. The 14-day holding time applies only to 1,2,3-Trichloropropane.

11.3INITIAL CALIBRATION – Instrument calibration (See Appendix 3 for details) criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the GC/ECD is capable of producing data within a range that is linear. The ICAL includes initial verification (ICV) of the ICAL with a second source standard for USACE and AFCEE projects (See Appendix 13 for details).

11.3.1 Compounds – Check to see if any other compounds are outside the criteria of 20% or less for %RSD. If a compound is outside the 20% criteria, check to see if linear regression was used. The limit for "r" used in linear regression is 0.990.

11.3.2 ACTION – Positive results for compounds not meeting the criteria may need to be qualified as estimated (J) by the user (client). Report detects

and non-detects without qualifiers and mention these outliners in the case narrative.

11.4 CONTINUING CALIBRATION – The calibration verification consists of two parts: (a) Initial calibration verification (ICV), which is performed using a second source standard to check on the ICAL standard; and (b) Continuing calibration verification (CCV), which is used to check that the instrument is operating under the linear range of the initial calibration. The CCV is also used to check the stability of the machine and to verify that the instrument is still operating under the linear range of the initial calibration and has remained stable every 12 hours.

11.4.1 Compounds – Check to see if the %D for any compounds are outside the criteria of 15% or less. Check to see that the mean %D for all the target compounds is <15%.

11.4.2 ACTION – Positive results for compounds not meeting the criteria may be qualified as estimated (J) by the user (client). Report non-detects without qualifiers and mention these outliners in the case narrative.

11.5 METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Sample data qualification to alert blank contamination is performed by the laboratory. Sample data qualification based on all the blank results (laboratory MB, equipment blank, and other field blanks) is performed by the user (client). The processing of blanks helps eliminate reporting of false positives in project reports.

11.5.1 Method blanks should be free of all target anaytes and other interfering non-target analytes. To be acceptable, blank results should not exceed one half of the applicable MQL for any compound.

11.5.2 Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples. If levels greater than one half the MQLs are present, inspect and correct the problem. Reanalyze all associated samples. Contact client if other criteria are applicable. The only exception may be quick turn around projects.

11.5.3 ACTION – All positive results are reported with a “B” code, if contaminants are found in the method blank.

11.5.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, then no action is taken.

11.5.5 ACTION – Any compound detected in the sample which were also detected in associated method blank at levels greater than one half the MQLs should be re-extracted unless other client criteria are applicable.

11.5.6 ACTION – If contaminants resulting in GC/ECD peak saturation are present, all affected compounds are qualified as invalid (I).

11.6 SURROGATE RECOVERIES – Surrogates or system monitoring compounds are different structurally, but similar chemically to classes of target compounds. These surrogate compounds are added just prior to extraction of the samples and the percent recoveries of the surrogates are calculated and evaluated.

11.6.1 Verify that the recovery (See Appendix 9 for details) of the surrogate compound (1,2-Dibromopropane or DBP) is within QC limits (70% to 120% in water and 60 to 130% in soil). Flag outliers with an asterisk (*).

11.6.2 ACTION – When surrogate recoveries are outside control limits the sample must be re-analyzed. Reanalysis may be performed under dilution if original analysis should indicate high levels of target and non-target analytes. Reextraction and cleanup will be necessary if reanalysis does not resolve the problem. If surrogate recoveries are still outside the limits, report both sets of data and described the problem in the case narrative.

11.7 LABORATORY CONTROL STANDARD (LCS) – A LCS analysis is used to evaluate method competence in the absence of matrix effects. The LCS should be from a second source standard. The LCS may provide a mechanism to check method efficiency for target compounds without the sample matrix as a factor.

11.7.1 LCS and LCSD recoveries (See Appendix 8 for details) should be within QC limits (See Tables 1 and 2).

11.7.2 Check the results for the LCS recoveries to see if the results are within the acceptable range. Flag outliers with an asterisk (*).

11.7.3 ACTION – If deviations are noticed, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements. Check with the project manager about such requirements. No action may need to be taken on LCS and/or LCSD recovery data alone either to reanalyze samples and/or to qualify associated samples for most projects. Professional judgment may be used by the user in evaluating sample data qualification options in conjunction with other QC data for the project.

11.8 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike data (See Appendix 7 for details) are used to evaluate the precision and accuracy of the analytical method on project samples.

11.8.1 The MS/MSD percent recoveries sould be within QC limits (Tables 1 and 2).

11.8.2 Check the results for the MS/MSD recoveries. Flag outliers with an asterisk (*).

11.8.3 Perform one manual calculation using raw data and extraction sheet information.

11.8.4 ACTION – No action is taken on MS/MSD recoveries data alone to qualify all associated samples. A general rule is to have at least 90% of the target analytes within the control limits. Since matrix interference could

lead to lead to serious analytical problems, the supervisor should be consulted when significant outliers are noted. Document QC outliers for %recovery and RPD on the checklist and on client reports (*) and draw attention to the QC outliers on the case narrative. No action may be taken on MS/MSD recovery data alone either to reanalyze samples and/or to qualify associated samples either by the analyst or by the end user. Professional judgment may be used by the user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

11.9 TARGET COMPOUND IDENTIFICATION – Criteria for GC/ECD qualitative analysis is to minimize the number of erroneous positive or negative identifications of compounds.

11.9.1 Positive compounds must be within +/- 0.06 relative time (RRT) units of the standard RRT. The results should be >MDL or other threshold for reporting positives (e.g. LLR, RL) on both columns.

11.9.2 The RPD for sample amounts from the two columns for identified compounds should be within <40%. Document RPD calculations (See Appendix 6 for details) in Sample Result worksheets (Form-10 equivalents).

11.9.3 ACTION - If the RPD for positive results are <40%, report the result from the primary column without any flag.

11.9.4 ACTION - If the RPD is >40%, report the result from the primary column with a "J" flag. The result from the confirmation column may be reported with a "J" flag at the discretion of the analyst, if intererence is suspected for the compound of interest in the primary column.

11.9.5 ACTION – Professional judgement must be used when applying the criteria for GC/ECD analysis of target analytes. Take into account factors such as matrix effect and column bleed.

12.0 REFERENCES

12.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8011, 1,2-Dibromoethane and 1,2-Dibromo-3-chloropropane by Microextraction and Gas Chromatogrpahy, July 1992.

12.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.

12.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

12.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3500B, Organic Extraction and Sample Preparation, December 1996.

12.5 Methods for the Determination of Organic Compounds in Drinking Water, EPA/600/4-88-039, July 1991, Method 504, 1,2-Dibromoethane and 1,2-Dibromo-3-chloropropane in water by Microextraction and Gas Chromatogrpahy, Revision 1.0 (1986) and Revision 2.0 (1989).

12.6 Methods for the Determination of Organic Compounds in Drinking Water, EPA Method 504.1, 1,2-Dibromoethane (EDB), 1,2-Dibromo-3-chloropropane (DBCP), and 1,2,3-Trichloropropane (123TCP) in water by Microextraction and Gas Chromatogrpahy, 1993.

12.7 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3511, Organic compounds in water by microextraction, Revision 0, November 2002.

12.8 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3570, Microscale Solvent Extraction (MSE), Revision 0, November 2002.

12.9 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.

12.10 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

12.11 AML Standard Operating Procedures, Q-Water-RC.

12.12 AML Standard Operating Procedures, Q-WasteMgmt-RD.

12.13 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

12.14 AML Standard Operating Procedures, Q-Controlcharts-RC.

Table 1
Aqueous Sample MDL, LLR, MQL and QC Limits for LCS and MS

EDB and DBCP Analysis by Microextraction and GC/ECD													
Analytical Management Laboratories													
Preparation Method:	8011								Analyst:		RRH/KLL		
Analysis Method:	8011								Matrix:		Water		
	MDL	**MDL**	**MDL**	**MDL**	**LLR**	**MQL**	**Ratio (RQ)**		**LCS Limits**		**MS Limits**		**RPD**
Compound	Spike	DB-5	DB-1701	Report	Report	Report	QL/DL	flag	Lower	Upper	Lower	Upper	Limit
	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L			**%R**	**%R**	**%R**	**%R**	%
EDB	0.017	0.0060	0.0095	**0.0095**	0.01	**0.057**	6.0		80	120	70	130	20
DBCP	0.017	0.0061	0.0036	**0.0061**	0.01	**0.057**	9.3		80	120	70	130	20
TCP									80	120	70	130	20
Criteria for the ratio (RD) of mean amount found to MDL: >1 but <5 for water													
Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.													
LCS and MS %R limits are from the USACE EM200-1-3, Appendix I or from AFCEE documents.													

Table 2
Soil/Sediment Sample MDL, LLR, MQL and QC Limits for LCS and MS

EDB and DBCP Analysis by Microextraction and GC/ECD													
Analytical Management Laboratories													
Preparation Method:	8011												
Analysis Method:	8011								Matrix:		Soil		
				MDL	**LLR**	**MQL**	**Ratio (RQ)**		**LCS Limits**		**MS Limits**		**RPD**
Compound				Report	Report	Report	QL/DL	flag	Lower	Upper	Lower	Upper	Limit
				ug/kg	ug/kg	ug/kg			**%R**	**%R**	**%R**	**%R**	%
EDB				**0.05**	0.10	**0.30**	6.0		70	130	60	140	30
DBCP				**0.03**	0.10	**0.30**	10.0		70	130	60	140	30
TCP									70	130	60	140	30
				Mean MQL/MDL ratio:			8.0						
Soil MDLs were estimated from aqueous MDLs and the dilution factor (5).													
Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.													
LCS and MS %R limits are from the USACE EM200-1-3, Appendix I or from AFCEE documents.													

Table 3
List of Appendices

Appendix Number	Appendix Topic
1	Chromatography Conditions
2	Retention Time Summary
3	Initial Calibration (ICAL) Summary (Form-6 equivalents) Instrument Printouts
4	Calibration Verification (ICV and CCV) Summary (Form-7 equivalents) Instrument Printouts and Standard Chromatograms
5	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
6	Sample Result Worksheets (Form-10 equivalents)
7	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
8	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
9	Surrogate Recovery Reports (Form-2 equivalents)
10	Method Detection Limit (MDL) Study Summary
11	QC Association Forms (Form-4 equivalents) Sample Preparation Logsheets Instrument Run Log
12	Sample Reports (Form-1 equivalents)
13	Method Quality Objectives for Method 8000B and/or 8011 (USACE, AFCEE, DoD QSM, etc.)

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. O-MADEP-EPH-RC

EXTRACTABLE PETROLEUM HYDROCARBONS (EPH) By GC/FID Using the Massachusetts Department of Environmental Protection (MADEP) Method

(Based on EPA SW846-8015B and 8100 Methods)

October 28, 2005

APPROVED:

_____ _____
Approved By: Kendall Lindquist, B.S., MBA Date
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EXTRACTABLE PETROLEUM HYDROCARBONS (EPH) By GC/FID Using the Massachusetts Department of Environmental Protection (MADEP) Method

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) is used to determine extractable petroleum hydrocarbons (EPH) in a variety of matrices using the Revision 0, January 1998 procedure developed by the Massachusetts Department of Environmental Protection (MADEP) for the State underground storage tanks (UST) program. This SOP and the underlying MADEP-EPH methods, which are applicable to water, soil, sediment and other types of samples is based on guidance from EPA SW-846 Method 8015B, Method 8100 and Method 8000B. The terms, SOP (developed by AML), procedure, and method (developed by MADEP) are used interchangeably in this document.

1.1 This method is designed to measure the collective concentrations of extractable aliphatic and aromatic petroleum hydrocarbons in water and soil. Extractable aliphatic hydrocarbons are collectively quantitated within two ranges: C9 through C18, and C19 through C36. Extractable aromatic hydrocarbons are collectively quantitated within the C11 through C22 range. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 150 °C and 500 °C.

1.2 This method is based on a solvent extraction, silica gel solid-phase extraction/fractionation process (SPE), and gas chromatography (GC) analysis using a flame ionization detector (FID). This procedure should be used by, or under the supervision of, analysts experienced in extractable organics analysis. Analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool.

1.3 This method is designed to complement and support the toxicological approach developed by the Massachusetts Department of Environmental Protection to evaluate human health hazards that may result from exposure to petroleum hydrocarbons.

1.4 This method is also able to measure the individual concentrations of Target Polynuclear Aromatic Hydrocarbons (PAH) Analytes, including Diesel PAH Analytes, in water and soil. The use of this method to quantitate these analytes is optional, and the Method Detection Limits (MDLs) for some of these PAH compounds in water are greater than the notification and/or cleanup standards specified in the Massachusetts Contingency Plan for sites located in drinking water resources areas. In cases where it is necessary to demonstrate compliance with these standards, the use of a GC/MS method in the SIM mode and/or HPLC methodology may be necessary. A separate AML SOP based on 8270-SIM should be used to obtain lower detection limits for these compounds, when necessary.

1.5 The fractionation step described in this method may be eliminated to allow for the determination of a Total Petroleum Hydrocarbon (TPH) value, and/or obtain qualitative "fingerprinting" information. While TPH provides little information on the chemistry, toxicity,

or environmental fate of petroleum mixtures, it may be a cost-effective screening tool in cases where a relatively low concentration of contamination is suspected.

1.6 Petroleum products suitable for evaluation by this method include kerosene, fuel oil #2, fuel oil #4, fuel oil #6, diesel fuel, jet fuel, and certain lubricating oils. This method, in and of itself, is not suitable for the evaluation of gasoline, mineral spirits, petroleum naphthas, or other petroleum products, which contain a significant percentage of hydrocarbons lighter than C9. This method, in and of itself, is also not suitable for the evaluation of petroleum products, which contain a significant percentage of hydrocarbons heavier than C36.

1.7 The AML Method Quantitation Limit (MQL), which is synonymous with the MADEP Reporting Limit (RL) for each of the collective aliphatic and aromatic fractional ranges is approximately 2- to 10-mg/kg in soil, and approximately 50- to 100-µg/L in water. The MQL (RL) of this method for TPH is approximately 10-mg/kg in soil and approximately 100-µg/L in water. The MQL (RL) of this method for the Target PAH Analytes is compound-specific, and ranges from approximately 0.5- to 1.0-mg/kg in soil, and 1- to 5- µg/L in water.

1.8 This method includes a data adjustment step to subtract the concentration of Target Analytes from the concentration of C11 through C22 Aromatic Hydrocarbons. This step may be taken by the laboratory or the data user.

1.9 Data generated using this SOP should be reported using the AML reporting format, which is based on the recommended MADEP format.

1.10 Like all GC procedures, this method is subject to a "false positive" bias in the reporting of Target PAH Analytes, in that non-targeted hydrocarbon compounds eluting or co-eluting within a specified retention time window may be falsely identified and/or quantitated as a Target or Diesel PAH Analyte. While cleanup procedures specified in this method to segregate aliphatic and aromatic fractions will serve to mitigate this concern, confirmatory analysis by dissimilar columns, gas chromatography/mass spectrometry (GC/MS) analysis, or other suitable technique is recommended in cases where a target PAH analyte reported by this method exceeds an applicable reporting or cleanup standard, and/or where coelution of a non-targeted hydrocarbon compound is suspected.

1.11 The first draft of the MADEP method was evaluated by two inter-laboratory “Round Robin” testing programs. In the final evaluation effort, participating laboratories were provided (single-blind) sand samples spiked with a #2 fuel oil, and a “real world” groundwater sample contaminated by a highly weathered fuel oil. Laboratory proficiency was evaluated using a Z-score approach. Data received from 23 laboratories performing the method without significant modifications are summarized in Table 1. Laboratory and method performance on the water sample were adversely impacted by the relatively low concentrations of the aliphatic fractions (due to the low solubilities of these hydrocarbons in real world samples), and by leaching of naphthalenes into the aliphatic extract during fractionation. Improvements incorporated into the final Revision 0 of the MADEP method are expected to mitigate problems of this nature and significantly improve overall method performance.

1.12 The underlying MADEP procedure is a performance-based method. Modifications to this method are permissible, provided that adequate documentation exists, or has been developed, to demonstrate an equivalent or superior level of performance. MADEP encourages methodological innovations which (a) better achieve method and/or data quality objectives, (b) increase analytical precision and accuracy, (c) reduce analytical uncertainties and expenses, and/or (d) reduce the use of toxic solvents and generation of hazardous wastes. Laboratories that modify this method must achieve all required performance and acceptance standards, and must have on file a Standard Operating Procedure which thoroughly describes the revised or alternative method and documentation which demonstrates an equivalent or superior level of performance. All significant modifications to the method must be disclosed and described on the data report form, as detailed in Section 11.3. Minor modifications made by AML to the MADEP procedure are described in this SOP document.

1.13 Additional information and details on the MADEP VPH/EPH approach, and the results of inter-laboratory "Round Robin" evaluations of this method, are available on the World Wide Web at http://www.magnet.state.ma.us/dep/bwsc/pubs.htm.

2.0 SUMMARY OF METHOD

2.1 A sample submitted for EPH analysis is extracted with methylene chloride, dried over sodium sulfate, solvent exchanged into hexane, and concentrated in a Kuderna-Danish apparatus. Sample cleanup and separation into aliphatic and aromatic fractions is conducted using commercially available silica gel cartridges or self-packed silica gel columns. The two extracts produced are then re-concentrated to final volumes of 1-mL each (i.e., an aliphatic extract and an aromatic extract). The extracts are then separately analyzed by a capillary column gas chromatograph equipped with a flame ionization detector. The resultant chromatogram of aliphatic compounds is collectively integrated within the C9 through C18 and C19 through C36 ranges. The resultant chromatogram of aromatic compounds is collectively integrated within the C11 through C22 range, and is (optionally) used to identify and quantitate individual concentrations of Target PAH Analytes.

2.2 Average calibration factors or response factors determined using an aliphatic hydrocarbon standard mixture is used to calculate the collective concentrations of C9 through C18 and C19 through C36 aliphatic hydrocarbons. An average calibration factor or response factor determined using a PAH standard mixture is used to calculate a collective C11 through C22 aromatic hydrocarbon concentration. Calibration factors or response factors determined for individual components of the PAH standard mixture are also used to calculate individual concentrations of Target PAH Analytes.

2.3 This method is suitable for the analysis of waters, soils, and sediments.

2.4 This method is based on (1) USEPA Methods 8000, 8100, and 3630, SW-846, "Test Methods for Evaluating Solid Waste", 3rd Edition, 1986; (2) Draft "Method for Determination of Diesel Range Organics", EPA UST Workgroup, November, 1990; and (3) "Method for Determining Diesel Range Organics", Wisconsin Department of Natural Resources, PUBL-SW-141, 1992.

3.0 DEFINITIONS

3.1 **Aliphatic Hydrocarbon Standard** is defined as a 14 component mixture of the normal alkanes listed in Table 2. The compounds comprising the Aliphatic Hydrocarbon Standard are used to (a) define and establish windows for the two aliphatic hydrocarbons ranges, and (b) determine average chromatographic response factors that can in turn be used to calculate the collective concentration of aliphatic hydrocarbons in environmental samples within those hydrocarbon ranges.

3.2 **Analytical Batch** is defined as a group of field samples with similar matrices which are processed as a unit. For Quality Control purposes, if the number of samples in such a group is greater than 20, then each group of 20 samples or less are defined as separate analytical batches.

3.3 **Aromatic Hydrocarbon Standard** is defined as a 17 component mixture of the polynuclear aromatic hydrocarbons (PAHs) listed in Table 3. The compounds comprising the Aromatic Hydrocarbon Standard are used to (a) define the individual retention times and chromatographic response factors for each of the PAH analytes listed in Table 3, (b) define and establish the window for the C11 through C22 Aromatic Hydrocarbon range, and (c) determine an average chromatographic response factor that can in turn be used to calculate the collective concentration Extractable Petroleum Hydrocarbons of aromatic hydrocarbons in environmental samples within the C11 through C22 hydrocarbon range.

3.4 **C9 through C18 Aliphatic Hydrocarbons** are defined as all aliphatic hydrocarbon compounds eluting from n-nonane (n-C9) to just before n-nonadecane (n-C19).

3.5 **C19 through C36 Aliphatic Hydrocarbons** are defined as all aliphatic hydrocarbon compounds eluting from n-nonadecane (n-C19) through n-hexatriacontane (n-C36).

3.6 **C11 through C22 Aromatic Hydrocarbons** are defined as all aromatic hydrocarbon compounds eluting from naphthalene through Benzo (g, h, i) Perylene, excluding Target PAH Analytes.

3.7 **Calibration Check Standard (CCV)** is defined as a calibration standard used to periodically check the calibration state of an instrument. The calibration check standard is prepared from the same stock standard solution as calibration standards, and is generally one of the mid-level range calibration standard dilutions.

3.8 **Calibration Standards used for initial calibration (ICAL)** are defined as a series of standard solutions prepared from dilutions of a stock standard solution, containing known concentrations of each analyte and surrogate compound of interest.

3.9 **Diesel PAH Analytes** are defined as naphthalene, 2-methylnaphthalene, phenanthrene, and acenaphthene, and are a subset of Target PAH Analytes. For most sites contaminated by a release of (only) diesel or #2 fuel oil, Diesel PAH Analytes will be the only Target PAH Analytes of interest.

3.10 **Extractable Petroleum Hydrocarbons (EPH)** are defined as collective fractions of hydrocarbon compounds eluting from n-nonane to n-hexatriacontane, excluding Target PAH Analytes. EPH is comprised of C9 through C18 Aliphatic Hydrocarbons, C19 through C36 Aliphatic Hydrocarbons, and C11 through C22 Aromatic Hydrocarbons.

3.11 **Field Duplicates** are defined as two separate samples collected at the same time and location under identical circumstances and managed the same throughout field and laboratory procedures. Analyses of field duplicates give a measure of the precision associated with sample collection, preservation and storage, as well as laboratory procedures.

3.12 **Fractionation Surrogate Standards** are compounds that are spiked into the sample extract immediately prior to fractionation, in order to determine if significant quantities of naphthalene or substituted naphthalenes are being stripped into the aliphatic extract.

3.13 **Laboratory Fortified Blank (LFB) or Laboratory Control Sample (LCS)** is defined as a reagent water blank or clean sand blank fortified with a matrix spiking solution. The LFB sample can be analyzed in duplicate **(LFBD or LCSD)** to obtain method precision and accuracy data.

3.14 **Laboratory Duplicates** are defined as split samples taken from the same sampling container and analyzed separately with identical procedures. The analysis of laboratory duplicates give a measure of the precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

3.15 **Laboratory Fortified Matrix (LFM) or Matrix Spike (MS) Sample** is defined as an environmental sample, which has been spiked with a matrix spiking solution containing known concentrations of method analytes. The LFM (MS) sample is treated and analyzed exactly as other samples, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentration of analytes in the sample matrix must be determined through the separate analyses of a laboratory or field duplicate, and the measured values in the LFM sample corrected for background concentrations. The LFM sample can be analyzed in duplicate **(LFMBD or MSD)** to obtain matrix precision and accuracy data.

3.16 **Laboratory Method Blank (MB)** is defined as an aliquot of reagent water or clean sand spiked with a surrogate standard. The laboratory method blank is treated exactly as a sample, exposed to all glassware, solvents, reagents, and equipment. A laboratory method blank is analyzed with every batch of samples, to determine if method analytes or other interferences are present in the laboratory environment, reagents, or equipment.

3.17 **Matrix Spiking Solution** is defined as a solution prepared independently from the calibration standards, containing known concentrations of method analytes.

3.18 **System Solvent Blank** is defined as an aliquot of a method solvent (e.g., hexane or methylene chloride, pesticide grade or better) that is directly injected into the GC system. The purpose of the System Solvent Blank is to determine the level of noise and baseline rise attributable solely to the GC system, in the absence of any other analytes or system

contaminants.

3.19 **Surrogate Standards** are compounds spiked into all samples, blanks, and matrix spikes to monitor the efficacy of sample extraction, chromatographic, and calibration systems.
.
3.20 **Target PAH Analytes** are defined as the 17 polynuclear aromatic hydrocarbon (PAH) compounds listed in Table 3.

3.21 **Total Petroleum Hydrocarbons (TPH)** are defined as the collective concentration of all hydrocarbon compounds eluting from n-nonane to n-hexatriacontane, excluding Target PAH Analytes. TPH is equivalent to the summation of C9 through C18 Aliphatic Hydrocarbons, C19 through C36 Aliphatic Hydrocarbons, and C11 through C22 Aromatic Hydrocarbons.

3.22 **Unadjusted C11 through C22 Aromatic Hydrocarbons** are defined as all aromatic hydrocarbon compounds eluting from naphthalene through Benzo (g, h, i) Perylene.

3.23 **Unadjusted TPH** is defined as the collective concentration of all hydrocarbon compounds eluting from n-nonane to n-hexatriacontane.

4.0 INTERFERENCES

4.1 Method interferences are reduced by washing all glassware with hot soapy water and then rinsing with warm tap water, acetone, and methylene chloride.

4.2 High purity reagents must be used to minimize interference problems.

4.3 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is analyzed, it must be followed by the analysis of a system solvent blank to check for cross-contamination.

4.4 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interference will vary considerably from one source to another depending upon the nature and diversity of the site being sampled. A silica gel SPE cleanup procedure is used to overcome many of these interferences, but some samples may require additional cleanup approaches which are beyond the scope of this method.

4.5 Certain organic compounds not associated with releases of petroleum products, including chlorinated hydrocarbons, phenols, and phthalate esters, will be quantitated as Extractable and Total Petroleum Hydrocarbons. If necessary and/or desirable, additional sample cleanup and/or analytical procedures may be employed to minimize or document the presence of such compounds.

4.6 The leaching of plasticizers and other compounds has been observed from commercially available silica gel cartridges used to fractionate EPH sample extracts. Concerns of this nature must be continuously monitored and documented by analysis of Laboratory Method Blanks.

4.7 Because of their weakly polar nature, naphthalene and substituted naphthalenes are readily fractionated into the aliphatic extract if excessive amounts of hexane are used to elute the silica gel cartridge/column. Because these compounds constitute a significant percentage of the water-soluble fraction of fuel oils, this occurrence is especially problematic in the analysis of water samples. For this reason, the method requires the use of fractionation surrogate standards with properties similar to naphthalene to monitor and document problems of this nature.

5.0 HEALTH AND SAFETY ISSUES

5.1 The toxicity and carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available.

5.2 The laboratory is responsible for maintaining a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method.

5.3 A reference file of material safety data sheets (MSDS) should also be made available to all personnel involved in the chemical analysis.

6.0 APPARATUS AND MATERIALS

6.1 The following glassware is used for this method:

- 6.1.1 1-L amber glass bottles
- 6.1.2 4 oz. (120-mL) amber glass wide-mouth jars
- 6.1.3 Vials:
  - 6.1.3.1 Auto sampler: 2-mL glass vials with Teflon-lined rubber crimp caps
  - 6.1.3.2 10-mL vials with Teflon-lined caps
- 6.1.4 Glass funnels
- 6.1.5 2-L Separatory funnels with Teflon stopcock
- 6.1.6 Kuderna-Danish apparatus including 10-mL graduated concentrator tube, 500-mL Evaporative flask, & 3-ball Snyder column
- 6.1.7 250-mL Erlenmeyer flasks
- 6.1.8 Disposable pipets: Pasteur
- 6.1.9 25-mL graduated cylinder
- 6.1.10 1-Liter graduated cylinder
- 6.1.11 100-mL beakers
- 6.1.12 Class “A” volumetric flasks: 10, 25, 50 and 100-mL
- 6.1.13 Class “A” volumetric pipets: 1, 5 or 10-mL

6.2 Analytical balance: An analytical balance capable of accurately weighing 0.0001 g must be used for weighing standards. A top-loading balance capable of weighing to the nearest 0.1 g must be used for weighing soil samples.

6.3 An air or nitrogen blow-down apparatus, or equivalent sample concentration apparatus, is

required to concentrate extracts.

6.4 **Gas Chromatography**

6.4.1 Gas Chromatograph: An analytical system complete with temperature programmable gas chromatograph (Agilent (HP) 5890) for use with capillary columns is required. The data station must be capable of storing and reintegrating chromatographic data (Agilent Chemstation) and must be capable of determining peak areas using a forced baseline projection.

6.4.2 Recommended Column: 30-m long x 0.32-mm I.D., 0.25-µm film RTX5 column (Restek Corp.) or equivalent. This column will allow for the adequate resolution of alkanes from n-C9 to n-C36. This column will also resolve the Target PAH Analytes listed in Table 3. The laboratory uses a 30-m DB-5 column, which provides adequate separation for the PAH analytes.

6.4.3 Detector: A Flame Ionization Detector (FID) is required. The Agilent GC is equipped with an Agilent FID detector.

6.4.4 Auto sampler: An auto sampler capable of making 1 to 4 µL injections is recommended. The Agilent GC is equipped with an Agilent (HP) 7673 autosampler.

6.5 Water bath: heated with a concentric ring cover, capable of temperature control (± 2°C). The bath should be used in a hood.

6.6 Microsyringes: 10-µL, 100-µL, 250-µL, 500-µL, 1000-µL

6.7 Boiling Chips

6.8 Soxhlet, Soxtec (two Tecator Soxtec-HT Model-1046 service units and three Model-1043 extraction units) or alternative extraction apparatus should be used for sample preparation.

6.9 Drying oven

6.10 SPE extraction manifold (Supelco) with 12 positions for sample extraction/cleanup, equipped with a Gast Model 2Z866 vacuum pump, vacuum control, and stopcocks for controlling fluid flow through the SPE columns.

6.11 A Tekmar Sonic Disruptor equipped with dual horns, and required pulsing and power capabilities will provide an alternate sample extraction mechanism for soil and sediment samples.

7.0 REAGENTS AND STANDARDS

7.1 **Reagents**

7.1.1 Reagent Water: organic free water (ASTM Type I reagent grade water).

7.1.2 Solvents: hexane, methylene chloride, and acetone; pesticide grade or better. Store away from other solvents.

7.1.3 Sodium sulfate: (ACS) granular, anhydrous. Purify by heating at 400°C for 4 hours in a shallow tray.

7.1.4 Ottawa and/or masonry sand: free of extractable petroleum hydrocarbons.

7.1.5 Silica Gel (5- to 10-grams), either prepared/packed by the laboratory, or purchased in 5-g/20-mL cartridges from a commercial vendor (Supelco LC-SI, catalogue # 57133). Smaller (1-g/6-mL) cartridges (J.T. Baker, BAKERBOND SPE silica gel (SiOH) disposable extraction columns, catalogue # 7086-07) may be used with water samples that are relatively clean. Silica gel prepared and packed by the laboratory should be activated at 130 °C for at least 16 hours, and heated to 150- to 160- °C for several hours before use. ***NOTE:*** Leaching of plasticizers and other compounds has been observed from commercially prepared silica gel cartridges, and must be monitored and documented by analyses of Laboratory Method Blanks.

7.2 **Stock Standard Solutions**

Prepare stock standard solutions at approximately 1000-ng/µL, or purchase as certified solutions.

7.2.1 Aromatic Hydrocarbon Standard: The Aromatic Hydrocarbon Standard consists of the 17 PAH compounds listed in Table 3, a surrogate compound, and fractionation surrogate compounds. Purchase EPH Aromatic Hydrocarbon Standard (MA) that contains these analytes (at 1000-µg/mL in methylene chloride) from Ultra Scientific (catalogue # SMA-300).

7.2.2 Aliphatic Hydrocarbon Standard: The Aliphatic Hydrocarbon Standard consists of the 14 normal alkanes listed in Table 2 and a surrogate compound. Purchase EPH Aliphatic Hydrocarbon Standard (MA) that contains these analytes (at 1000-µg/mL in hexane) from Ultra Scientific (catalogue # SMA-310).

7.2.3 Petroleum Reference Standard: The Petroleum Reference Standard consists of an API or commercial diesel fuel. Purchase Diesel Standard that contains this product (at 1000-µg/mL in ….) from Ultra Scientific (catalogue # ……..).

7.3 **Surrogate Standards**

7.3.1 Surrogate standards are used to monitor the efficacy of sample extraction, chromatographic, and calibration systems.

7.3.2 The recommended surrogate standards are chloro-octadecane (COD, available from Restek Corporation, Bellefonte, PA) and ortho-terphenyl (OTP, available from EM Sciences, Gibbstown, NJ). Purchase EPH Surrogate Spiking Solution (MA) that contains these two compounds (at 2000-µg/mL in acetone) from Ultra Scientific (catalogue # ISM-580).

7.3.3 The surrogate standard COD can be prepared by accurately weighing approximately 0.0100 g of pure material in a 10-mL volumetric flask. Dissolve the material in hexane. This solution is added to the Aliphatic Hydrocarbon standard.

7.3.4 The surrogate standard OTP can be prepared by accurately weighing approximately 0.0100 g of pure material in a 10-mL volumetric flask. Dissolve the material in methylene chloride. This solution is added to the Aromatic Hydrocarbon standard.

7.3.5 ***Surrogate Spiking Solution:*** The recommended surrogate spiking solution is comprised of a mixture of the COD and OTP surrogate standards. Prepare a surrogate spiking solution, which contains the surrogate standards at a concentration of 40-ng/µL (40-µg/mL) in acetone by diluting 2.0-mL of the 2000-µg/mL standard (Section 7.3.2) to 100-mL with acetone. Each sample, blank, and matrix spike is fortified with 1.0-mL of

the surrogate spiking solution. The use of higher concentrations is permissible, and may be necessary, when spiking highly contaminated samples.

7.4 **Fractionation Surrogate Standards**

7.4.1 The fractionation surrogate standards are added to the sample (hexane) extract just prior to fractionation. The purpose of the fractionation surrogate standards is to monitor the efficacy of the fractionation process, and ensure that unacceptable quantities of naphthalene and substituted naphthalenes are not being stripped into the aliphatic extract.

7.4.2 The recommended fractionation surrogate standards are 2-Fluorobiphenyl and 2-Bromonaphthalene. Alternative fractionation surrogate compounds are permissible, provided that a demonstration is made that such compounds exhibit polarities/fractionation properties similar to naphthalene.

7.4.3 The fractionation surrogate standards are prepared by accurately weighing approximately 0.0100 g of pure material in a 10-mL volumetric flask. Dissolve the material in Methylene Chloride. Purchase EPH Fractionation Surrogate Standard Mixture (MA) that contains these two compounds (at 2000-µg/mL in methylene chloride) from Ultra Scientific (catalogue # ISM-650). This solution is added to the Aromatic Hydrocarbon standard.

7.4.4 ***Fractionation Surrogate Spiking Solution:*** The recommended fractionation surrogate spiking solution is comprised of 2-Fluorobiphenyl and 2-Bromonaphthalene prepared in hexane at concentrations of 40-ng/µL (40-µg/mL) by diluting 2.0-mL of the 2000-µg/mL standard (Section 7.4.3) to 100-mL with hexane. An aliquot of 1-mL of the fractionation surrogate spiking solution is added to the 1 mL EPH sample extract prepared in accordance with the provisions of Sections 9.1.1, 9.1.2, and 9.1.3. Alternative concentrations/volumes of the fractionation surrogate spiking solution are permissible.

7.5 **Internal Standard**

The recommended internal standard is 5-alpha-androstane (EM Sciences, Gibbstown, NJ). Purchase a solution that contains this compound (at 2000-µg/mL in methylene chloride) from Ultra Scientific (catalogue # IST-500). Use of an internal standard for this method was evaluated by AML and it was concluded that external standard calibration is a preferable option.

7.6 **Matrix Spike Standard**

7.6.1 According to the MADEP method, five or more analytes from each analyte group (i.e., aromatic and aliphatic hydrocarbons) can be selected for use in a matrix spiking solution, which is prepared independently from the calibration standards.

7.6.2 The MADEP-recommended spiking solution consists of C9, C14, C19, C20, and C28 normal alkanes, and the following PAH compounds: naphthalene, acenaphthene, anthracene, pyrene and chrysene. The MS spiking solution is prepared in acetone at concentrations of 50-ng/µL (50-µg/mL). The AML spiking solution should contain all the target compounds at the specified concentration. Prepare the ***AML MS spiking solution*** by diluting 500-µL of the 1000-µg/mL EPA Aromatic Hydrocarbon Standard (Section 7.2.1) and 500-µL of the 1000-µg/mL EPA Aliphatic Hydrocarbon Standard (Section 7.2.2) to 10-mL with acetone.

7.6.3 The samples selected as the matrix spike are fortified with 1.0-mL of the matrix

spiking solution.

7.7 **Fractionation Check Solution**

7.7.1 The Fractionation Check Solution is used to monitor the fractionation efficiency of the silica gel cartridge/column, and establish the optimum volume of hexane needed to sufficiently elute aliphatic hydrocarbons, but not strip aromatic hydrocarbons.

7.7.2 Prepare a Fractionation Check Solution in hexane containing 200-ng/µL of the Aliphatic Hydrocarbon standard (C9-C36 alkanes) and 200-ng/µL of the Aromatic Hydrocarbon standard (targeted PAH analytes). The final solution will contain 14 alkanes and 17 PAHs at concentrations of 200-ng/µL each. Alternative concentrations are permissible.

8.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 Aqueous samples are collected in 1-liter amber glass bottles with Teflon-lined screw caps. Provide two (2) 1-liter bottles for each sample requiring this analysis

8.2 Soil and sediment samples are collected in 4-Oz. (120 mL) amber wide-mouth glass jars with Teflon-lined screw caps. Provide one (1) 4-Oz. jars for each sample requiring this analysis.

8.3 Aqueous samples must be preserved at the time of sampling by the addition of a suitable acid to reduce the pH of the sample to less than 2.0. This may be accomplished by the addition of 5-mL of 1:1 HCl to a 1 liter sample. The use of alternative acids is permissible. Following collection and addition of acid, the sample must be cooled to 4°C.

8.4 Soil and sediment samples must be cooled to 4°C immediately after collection.

8.5 A chain of custody form must accompany all aqueous, soil and sediment samples, documenting the time and date of sampling and any preservative additions.

8.6 **Aqueous samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction.** ***NOTE:*** *The EPA holding time for the extraction of organics from unpreserved samples (7-days) and the EPA holding time for the extraction of TPH from acid-preserved samples (28-days) are not applicable to the MADEP EPH method.*

8.7 **Soil and sediment samples must be extracted within 7 days of collection, and analyzed within 40 days of extraction.** ***NOTE:*** *The EPA holding time for the extraction of organics from soil samples (14-days) is not applicable to the MADEP EPH method.*

8.8 Table 4 contains a summary of sample collection, preservation, and holding times.

9.0 PROCEDURE

9.1 **Sample Preparation**

Samples are extracted using methylene chloride, and solvent-exchanged into hexane. The recommended extraction procedure for water samples is a separatory funnel liquid-liquid extraction technique based upon SW-846 Method 3510A. For soil or sediment samples, use of a

Soxhlet or Soxtec technique is recommended. Alternative extraction procedures are acceptable, provided that the laboratory can document acceptable performance. During the Round Robin evaluations conducted on this method, the use of a sonication extraction procedure appeared to provide acceptable performance for freshly-spiked sand samples.

9.1.1 Water Extraction

9.1.1.1 Mark the meniscus on the 1-liter sample bottle (for later volume determination) and transfer it to a 2-liter separatory funnel. For blanks and quality control samples, pour 1 liter of reagent water into the separatory funnel. Add 1.0-mL of the surrogate spiking solution to all samples, blanks and matrix spikes. For samples selected for spiking, add 1.0-mL of the matrix spiking solution.

9.1.1.2 Check the pH of the sample with wide-range pH paper. Note the pH in the laboratory notebook. The pH of the sample need not be adjusted.

9.1.1.3 Add 60-mL methylene chloride to the sample bottle to rinse the inner walls of the container, and then add this solvent to the separatory funnel.

9.1.1.4 Seal and shake the separatory funnel vigorously for 1- to 2-minutes with periodic venting to release excess pressure. **NOTE: Methylene chloride creates excessive pressure very rapidly; therefore, venting should be done immediately after the separatory funnel has been sealed and shaken once.**

9.1.1.5 Allow the organic layer to separate from the water phase for a minimum of 5-minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the solvent extract in an Erlenmeyer flask.

9.1.1.6 Repeat the extraction two more times using additional 60-mL portions of solvent. Combine the three solvent extracts (Steps 9.1.1.3 to 9.1.1.5) in a 250-mL Erlenmeyer flask.

9.1.1.7 For sample volume determination add water to the sample bottle to the level of the meniscus previously marked then transfer this water to a graduated cylinder.

9.1.1.8 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporation flask.

9.1.1.9 Dry the extract by passing it through a glass powder funnel containing anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Rinse the Erlenmeyer flask, which contained the solvent extract, with 20- to 30-mL of methylene chloride and add it to the funnel to complete the quantitative transfer.

9.1.1.10 Add one or two clean boiling chips to the K-D flask and attach a three ball Snyder column. Pre-wet the Snyder column by adding about 1-mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80-90°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10- to 20-minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 1-mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10-minutes.

9.1.1.11 Exchange the methylene chloride with hexane by adding 50-mL of hexane to the

top of the Snyder column. Concentrate the extract to less than 10-mL, as described in Paragraph 9.1.1.10, raising the temperature of the water bath, if necessary, to maintain proper distillation.

9.1.1.12 Remove the Snyder column and evaporation flask from the 10-mL concentrator tube. Place the concentrator tube containing the hexane extract onto an air blow-down apparatus. Adjust the extract volume to 1-mL under a gentle stream of nitrogen or air. If the extract is highly colored, forms a precipitate, or stops evaporating, the final volume should be higher.

9.1.1.13 Add 1-mL of the fractionation surrogate spiking solution to the 1-mL hexane extract. Alternatively, add 20- to 50-ng each of the fractionation surrogate standards using a microliter syringe and concentrated spiking solution.

9.1.1.14 Record the sample preparation information for the extraction and concentration steps. At a minimum, record the date, sample laboratory number, sample volume, volume and concentration of added surrogates, fractionation surrogates, and matrix spike solutions, and any deviations or problems associated with the extraction of the samples.

9.1.1.15 The 2-mL extract is now ready to be cleaned and fractionated by silica gel SPE. If cleanup will not be performed immediately, transfer the extract to a Teflon-lined screw-cap vial, label, and refrigerate. If a TPH analysis is to be conducted, without fractionation, proceed to Section 9.1.5

9.1.1.16 For cleanup and fractionation, refer to Section 9.1.4.

9.1.2 ***Soil/Sediment Extraction using Soxtec Extraction***

9.1.2.1 Blend 10-g of the solid sample (**NOTE: Use slightly larger amount, up to 15-g maximum to minimize the effect of percent solids correction on detection limits)** with 10-g (or as needed to dry the sample) anhydrous sodium sulfate and place in an extraction thimble. **(NOTE: The quantity of sample and the drying agent should fit within the thimble).** The extraction thimble must drain freely for the duration of the extraction period. Add 1.0-mL of the surrogate spiking solution onto the sample. For samples selected for spiking, add 1.0-mL of the matrix spiking standard.

9.1.2.2 Place 70-mL of methylene chloride into a 100-mL Soxtec beaker containing 1 or 2 boiling chips. Attach the beaker to the Soxtec extractor and extract the sample for 4 hours.

9.1.2.3 Allow the extract to cool after the extraction is complete.

9.1.2.4 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporation flask. Alternatively, sample concentration may be accomplished in the Soxtec extractor.

9.1.2.5 Dry the extract by passing it through a glass powder funnel containing anhydrous sodium sulfate. Collect the dried extract in the K-D concentrator. Wash the Soxtec cup and sodium sulfate column with 100- to 125-mL of methylene chloride to complete the quantitative transfer.

9.1.2.6 Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1-mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80- to 90- °C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10- to 20-min. At the proper rate of distillation, the balls of the column will actively chatter, but the

chambers will not flood. When the apparent volume of liquid reaches 1-mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10-minutes.

9.1.2.7 Exchange the methylene chloride with hexane by adding 50-mL of hexane to the top of the Snyder column. Concentrate the extract as described in Paragraph 9.1.2.6, raising the temperature of the water bath, if necessary, to maintain proper distillation.

9.1.2.8 Remove the Snyder column and evaporation flask from the 10-mL concentrator tube. Place the concentrator tube containing the hexane extract onto an air blow-down apparatus. Adjust the extract volume to 1-mL under a gentle stream of nitrogen or air. If the extract is highly colored, forms a precipitate, or stops evaporating, the final volume should be higher.

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9.1.2.9 Add 1-mL of the fractionation surrogate spiking solution to the 1-mL hexane extract. Alternatively, add 20- to 50-ng each of the fractionation surrogate standards using a microliter syringe and concentrated spiking solution.

9.1.2.10 Record the sample preparation information for the extraction and concentration steps. At a minimum, record the date, sample laboratory number, sample volume, volume and concentration of added surrogates, fractionation surrogates, and matrix spike solutions, and any deviations or problems associated with the extraction of the samples.

9.1.2.11 The 2-mL extract is now ready to be cleaned and fractionated by silica gel SPE. If cleanup will not be performed immediately, transfer the extract to a Teflon-lined screw-cap vial, label, and refrigerate. If a TPH analysis is to be conducted, without fractionation, proceed to Section 9.1.5

9.1.2.12 For cleanup and fractionation, refer to Section 9.1.4.

9.1.3 Soil/Sediment Extraction by Soxhlet Extraction – This procedure is not described in detail in this SOP since AML uses an automated equipment for this extraction procedure as described in Section 9.1.2. Refer to the MADEP Method (Section 9.1.3.1 to 9.1.3.12) for details, when necessary.

9.1.4 Silica Gel Cleanup and Fractionation

The silica gel column cleanup and fractionation procedures implemented at AML should use the Supelco Extraction Manifold (Section 6.10) for greater control, sample throughput, and efficiency. Insert an adaptor to the SPE cartridge, connect them to the vacuum manifold, turn on the vacuum pump, and adjust flow rates through the column using the vacuum controller and the stop cock. ***NOTE: The Silica Gel Cleanup and Fractionation step is a critical yet highly sensitive procedure. Small changes in the volumes of eluting solvents, fractionation equipment, and/or fractionation techniques can significantly impact the proportion of hydrocarbons segregated in either the aliphatic or aromatic fractions. Considerable care and attention is required to ensure satisfactory results. See also additional notes that are shown in bold italics.***

9.1.4.1 Each sample fractionation requires 1-mL of sample extract. Because 2-mL of sample extract is available, two fractionations may be undertaken for each sample. (Alternatively, 0.5 mL of sample extract may be fractionated if 20- to 50-ng each of the fractionation surrogate standards were added to the 1-mL sample extract using a microliter syringe and concentrated spiking solution.) Refractionation would be necessary if problems are experienced during the initial fractionation effort, and/or if unacceptable recoveries are noted for the fractionation surrogate standards. The

extra volume of sample extract is also provided to facilitate initial (unfractionated) TPH screening of a sample, to obtain a GC/FID "fingerprint", and/or to determine whether sufficient total hydrocarbons are present to warrant fractionation and comparison to risk-based cleanup standards.

9.1.4.2 Silica gel is a regenerative adsorbent of amorphous silica with weakly acidic properties. It is produced from sodium silicate and sulfuric acid. Silica gel can be used for column chromatography and is used for separating analytes from interfering compounds of a different chemical polarity. Silica gel is also used to separate petroleum distillates into aliphatic and aromatic fractions.

9.1.4.3 A 5-g/20-mL commercially prepared Solid Phase Extraction (SPE) silica gel cartridge is recommended. Alternatively, the use of self-packed columns of activated silica gel may also be used. ***NOTE: Silica gel/cartridges which have become contaminated with moisture may perform erratically. Unused cartridges in an unsealed lot are prone to contamination by atmospheric moisture. To ensure satisfactory results, the silica gel/cartridges must not be overloaded with excessive mass of hydrocarbons. It is recommended that loading be limited to no more than 5-mg total hydrocarbons/gram silica gel; for a 1-mL extract fractionated on a 5-gram silica gel cartridge, this would equate to an extract loading of no greater than 25,000-µg/mL.***

9.1.4.4 If concerns exist over the presence of contaminants in the silica gel/cartridge, pre-rinse the column with 30-mL of methylene chloride, followed by at least 5 minutes of air drying by vacuum, to complete dryness.

9.1.4.5 Rinse the column with 30-mL of hexane, or 60-mL if pre-rinsed with methylene chloride per Section 9.1.4.4. Let the hexane flow through the column until the head of the liquid in the column is just above the column frit. Close the stopcock to stop solvent flow. Discard the collected hexane.

9.1.4.6 Load 1.0-mL of sample extract onto the column. Open the stopcock, and start collecting elutant immediately in a 25-mL volumetric flask labeled "aliphatics".

9.1.4.7 Just prior to exposure of the column frit to the air, elute the column with approximately 19-mL more hexane, so that a total of approximately 20-mL of hexane is passed through the column. ***NOTE: It is essential that "plug flow" of the sample extract be achieved through the silica gel cartridge/column. Hexane should be added in 1- to 2-mL increments or dropwise using a pipet, with additions occurring when the level of solvent drops to the point just prior to exposing the column frit to air. The use of a stopcock is mandatory. Care must be taken to ensure that the silica gel in uniformly packed in the column. The analyst must be cognizant of any channeling, streaking, or changes in the silica gel matrix during fractionation; if any of these occur, the procedure must be repeated with another 1-mL volume of sample extract. The amount of hexane used during fractionation is critical. Excessive hexane (as little as 0.5 mL) can cause significant elution of lighter aromatics into the aliphatic fraction. Insufficient hexane will cause low recoveries of the aliphatic fraction. The volume of hexane recommended (20-mL) may need to be adjusted for each lot of silica gel/cartridges to optimize extraction efficiencies and sample fractionation. See Section 10.3.4 for specifications on the use and evaluation of Fractionation Check Solutions.***

9.1.4.8 Following recovery of the aliphatic fraction, elute the column with 20-mL of methylene chloride and collect the elutant in a 25-mL volumetric flask. Label this fraction "aromatics".

9.1.4.9 Transfer the contents of the 25-mL volumetric flasks into labeled graduated concentrator tubes. Concentrate the extracts to a final volume of 1-mL each under a gentle stream of air or nitrogen from an air blow-down apparatus.

9.1.4.10 Transfer the final 1-mL extracts to labeled two-mL glass auto sampler vials with Teflon-rubber crimp caps. If appropriate, add an internal standard at the appropriate concentration.

9.1.5 Proceed with the analysis in accordance with Sections 9.2 through 9.5. Analyze all laboratory method blanks and QC samples under the same conditions as that used for samples.

9.1.6 If chromatographic responses exceed the linear range of the system, dilute the aliphatic and/or aromatic extract(s) and re-analyze.

9.1.7 ***If unacceptable recoveries are noted for the fractionation surrogate standards, repeat sections 9.1.4.4 through 9.1.4.10 with another 1-mL aliquot of the sample extract and re-analyze.***

9.1.8 Determination of Percent Moisture

9.1.8.1 Soil and sediment sample results must be reported on a dry-weight basis. A portion of sample for moisture determination should be weighed out at the same time as the portion used for hydrocarbon determination.

9.1.8.2 Immediately after weighing a sample for extraction, transfer approximately 5- to 10- g of the sample into a tared crucible. Dry this 5- to 10-g sample overnight at 105°C in an oven, and reweigh. Allow to cool in a desiccator before reweighing. Calculate the percent moisture of the sample using the equations provided in Section 9.6.3.

9.2 **GC Conditions**

9.2.1 Gas Chromatographic Conditions – The capillary column and the temperature programming should be optimized for the separation of PAH compounds and for eluting the high boiling hydrocarbons within a reasonable amount of time. The conditions shown below resulted in acceptable separation of the target analytes on a 30-m DB-5 column. Other columns and conditions may be employed without a modification of this document as long as they result in acceptable performance.

9.2.1.1 Oven Program: Set oven temperature to 60°C for 4 minutes, and then ramp it at 8°C/min to 300 °C and hold for 4 minutes. Set the maximum temperature for the oven at 325 °C or as required for the column. Total run time is 44 minutes.

9.2.1.2 Sample/auto sampler injection is 2-µL (1- to 4- µL is acceptable).

9.2.1.3 Gas Flows: The recommended carrier gas is helium.

9.2.1.3.1 Carrier gas Flow: approximately 2- to 3-mL/min.

9.2.1.3.2 Air: approximately 400-mL/min.

9.2.1.3.3 Hydrogen: approximately 35-mL/min.

9.2.1.3.4 Make up gas flow: approximately 30-mL/min.

9.2.1.4 Miscellaneous:

9.2.1.4.1 FID temperature, 280 °C (high temperatures, e.g. 315 °C may be employed if sensitivity for high boiling hydrocarbons is required)

9.2.1.4.2 Injection port temperature, 250 °C (high temperatures, e.g. 285 °C may be employed if sensitivity for high boiling hydrocarbons is required)

9.2.1.4.3 GC operated in splitless mode for 0.5-min.

9.2.1.4.4 Column head pressure, approximately 15.0-psi at 60 °C

9.2.1.4.5 Linear velocity, approximately 50-cm/sec

9.2.2 GC Maintenance

9.2.2.1 Capillary columns: Clean and deactivate the glass injection port insert or replace with a cleaned and deactivated insert.

9.2.2.2 Break off the first few inches, up to one foot, of the injection port side of the column.

9.2.2.3 Remove the column and solvent backflush according to the manufacturer's instructions.

9.2.2.4 Bake out the column at approximately 300 °C. If these procedures fail to eliminate a column degradation problem, it may be necessary to replace the column.

9.3 **GC Retention Time Windows**

9.3.1 Before establishing windows, make sure the GC system is within optimum operating conditions. Make three injections of the Aromatic Hydrocarbon and Aliphatic Hydrocarbon standard mixtures throughout the course of a 72-hr period. Serial injections over less than a 72-hr period may result in retention time windows that are too tight.

9.3.2 Calculate the standard deviation of the three absolute retention times for each individual component in the Aromatic Hydrocarbon standard, the Aliphatic Hydrocarbon standard, and all surrogate, fractionation surrogates, and internal standards.

9.3.3 Plus or minus three times the standard deviation of the absolute retention times for each standard should be used to define the retention time window. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

9.3.4 In those cases where the standard deviation for a particular standard is zero, the laboratory should substitute the standard deviation of a closely eluting structurally similar compound to develop a valid retention time window.

9.3.5 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. This data must be retained by the laboratory.

9.3.6 EPH retention time (RT) windows are defined as beginning 0.1 minutes before the RT of the beginning marker compound and ending 0.1 minutes after the RT of the ending marker compound, except for n-C19, which is both a beginning and ending marker compound for two different ranges. EPH marker compounds and windows are summarized in Table 5. ***NOTE: The C9 - C18 Aliphatic Hydrocarbon range ends immediately (0.01 min) before the elution of the n-C19 peak. The C19 - C36 Aliphatic Hydrocarbon range begins 0.01 before the elution of the n-C19 peak; therefore there is no overlap of the two ranges and the n-C19 peak is only included in the C19 - C36 Aliphatic Hydrocarbon range.***

9.3.7 If a TPH analysis is done, without fractionation, TPH retention time (RT) windows are defined as beginning 0.1 minutes before the RT of n-Nonane and ending 0.1 minutes after the RT of n-Hexatriacontane.

9.4 **Calibration**

Calibrate the GC system using either the external standard procedure (Section 9.4.1) or the internal standard procedure (Section 9.4.2).

9.4.1 External standard calibration procedure. This is the preferred procedure at AML for GC/FID analysis.

9.4.1.1 ***ICAL Standards Preparation*** - Prepare Aromatic and Aliphatic Hydrocarbon calibration standards at a minimum of five concentration levels by adding volumes of one or more stock standard solutions to volumetric flasks and diluting to volume with

methylene chloride and hexane, respectively. The surrogate OTP and the fractionation surrogate standards are added to the Aromatic Hydrocarbon Standard; the surrogate COD is added to the Aliphatic Hydrocarbon Standard. One of the calibration standards must be at the concentration of the Reporting Limit or MQL. The other concentrations must correspond to the expected range of concentrations found in real samples or should define the working range of the detector. Prepare ICAL standards at the following seven calibration levels: 10, 20, 50, 70, 100, 150, and 200-ng/µL (or µg/mL) for the individual components. The lowest concentration level (10-µg/mL) is required since it is used to define the MQL. The individual and collective concentrations of standard analytes within each hydrocarbon range for these recommended calibration levels are provided in Table 6.

9.4.1.2 Inject 2-µL of each calibration standard using the technique that will be used to introduce the actual samples into the gas chromatograph. Tabulate peak height or area responses (preferred mode of operation) against the mass injected. The results can be used to prepare a calibration curve for each analyte. Alternatively, the ratio of the response to the amount injected, defined as the calibration factor (CF), may be calculated for method analytes at each standard
concentration using Equation 1.

Equation 1: Calibration Factor

$$\text{Calibration Factor (CF)} = \frac{\text{Area of Peak}}{\text{Total concentration of standard } (\mu g/mL)}$$

If the percent relative standard deviation (%RSD) of the calibration factor is equal to or less than 25% over the working range for all analytes of interest, as determined using Equation 2, linearity through the origin can be assumed, and the average calibration factor can be used in place of a calibration curve.

Equation 2: Percent Relative Standard Deviation

$$\%RSD = \frac{\text{Standard Deviation of the CFs}}{\text{Mean of the CFs}} * 100$$

9.4.1.3 A collective calibration curve or factor must also be established for each hydrocarbon range of interest. To calculate the collective CF for C9-C18 Aliphatic Hydrocarbons, C19-C36 Aliphatic Hydrocarbons, and C11-C22 Aromatic Hydrocarbons, tabulate the summation of the peak areas of all components in that fraction (e.g., C9-C18, 6 components) against the total mass injected, using a forced baseline projection, and Equation 3. A listing of the collective concentrations of standards within each hydrocarbon range is provided in Table 6. ***Note: The area for the surrogates must be subtracted from the area of the range in which they elute (e.g., COD is subtracted from the C19 - C36 Aliphatic Hydrocarbon range).***

Equation 3: Range Calibration Factor

$$\text{Range CF} = \frac{\text{Total area of all component peaks}}{\text{Total Analyte Concentration for the range } (\mu g/mL)}$$

9.4.1.4 At a minimum, the working calibration curve or calibration factor must be verified on each working day, and after every 20 samples, by the injection of a mid-level calibration standard to verify instrument performance and linearity. If the percent difference (%D) for any analyte varies from the predicted response by more than ± 25%, as calculated using Equation 4, a new calibration curve must be prepared for that analyte or range. ***NOTE: This is a minor AML method modification to avoid confusion for the analysts since RPD is normally calculated for duplicates and %D (deviation from initial conditions) is calculated for calibration verification. This modification will strengthen the AML SOP by providing more weight to the initial condition.***

Equation 4: Percent Difference (%D)

$$RPD = \frac{CF_v - CF_i}{CF_i} * 100$$

where:
CF_i = Mean Calibration Factor from calibration curve.
CF_v = Calibration Factor from calibration check.

9.4.1.5 ***Linear Regression Method:*** The concentrations of specific analytes or hydrocarbon ranges in aqueous and non-aqueous samples may also be calculated from a calibration curve by use of linear regression analysis. When linear regression is used, the correlation coefficient (r) should be equal to or greater than 0.99. Linear regression may be appropriate for hydrocarbon ranges that exhibit baseline problems (e.g. C-19 to C-36) due to column bleed at high temperatures.

9.4.1.6 For TPH analysis, without fractionation, response factors are developed based upon the response of all 31 aliphatic and aromatic component standards.

9.4.2 Internal standard calibration procedure – This is an optional procedure that may be used by AML, if required.

9.4.2.1 The suggested internal standard for this method is 5-alpha-androstane (EM Science, Gibbstown, NJ).

9.4.2.2 Prepare Aromatic and Aliphatic Hydrocarbon calibration standards at a minimum of five concentration levels (Table 6) by adding volumes of stock standard solutions to volumetric flasks. The surrogate OTP and the fractionation surrogates are added to the Aromatic Hydrocarbon Standard; the surrogate COD is added to the Aliphatic Hydrocarbon Standard. To each calibration standard, add a known constant amount of an internal standard and dilute with an appropriate solvent.

9.4.2.3 Inject 2-µL of each calibration standard using the same technique that will be applied to the samples. Tabulate the peak height or peak area responses against the

concentration of each compound and internal standard. Calculate response factors (RF) for each individual compound using Equation 5. If the RF value over the working range is constant (< 25% RSD), the RF can be assumed to be invariant, and the average RF can be used for calculations.

Equation 5: Response Factor

$$RF = \frac{(A_s) * (C_{is})}{(A_{is}) * (C_s)}$$

where:
A_s = Area response for the analyte to be measured.
C_{is} = Concentration of the internal standard, ng/µL.
A_{is} = Area response for the internal standard.
C_s = Concentration of the analyte to be measured, ng/µL.

9.4.2.4 A collective response factor must also be established for each hydrocarbon range of interest. To calculate the collective RFs for C9-C18 Aliphatic Hydrocarbons, C19-C36 Aliphatic Hydrocarbons, and C11 - C22 Aromatic Hydrocarbons, tabulate the summation of the peak areas of all components in that fraction (e.g., C9-C18, 6 components) against their total concentration, using a forced baseline projection, and Equation 6. A listing of the collective concentrations of standards within each hydrocarbon range is provided in Table 6. ***Note: The area for the surrogates must be subtracted from the area of the range in which they elute (e.g., COD is subtracted from the C19-C36 Aliphatic Hydrocarbon range).***

Equation 6: Range Response Factor

$$Range\ RF = \frac{(A_s) * (C_{is})}{(A_{is}) * (C_s)}$$

A_s = Summation of peak areas of component standards (e.g., C9-C18 Aliphatic Hydrocarbons, 6 components).
C_{is} = Concentration of internal standard, ng/µL.
A_{is} = Response for the internal standard.
C_s = Total concentration of standards in the range, ng/µL. (See Table 6)

9.4.2.5 At a minimum, the RF must be verified on each working day, and after every 20 samples, by the injection of a mid-level calibration standard to verify instrument performance and linearity. If the percent difference (%D) for any analyte varies from the predicted response by more than ± 25%, as calculated using Equation 4, a new calibration curve must be prepared for that analyte or range. ***NOTE: This is a minor AML method modification to avoid confusion for the analysts since RPD is normally calculated for duplicates and %D (deviation from initial conditions) is calculated for calibration verification. This modification will strengthen the AML SOP by providing more weight***

to the initial condition.

9.4.2.6 The concentrations of specific analytes or hydrocarbon ranges in aqueous and non-aqueous samples may also be calculated from a calibration curve by the use of linear regression analysis.

9.4.2.7 For TPH analysis, without fractionation, response factors are developed based upon the response of all 31 aliphatic and aromatic component standards.

9.5 **GC Analysis**

9.5.1 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample extracts interspersed with blanks and QC samples. The sequence ends when the set of sample extracts has been injected or when qualitative and/or quantitative QC criteria are exceeded.

9.5.2 Aliphatic and aromatic extracts are introduced into the gas chromatograph by direct injection.

9.5.3 Inject 2-µL of the sample extract using the solvent flush technique. Smaller volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 µL and the resulting peak size in area units or peak height.

9.5.4 Establish daily retention time windows for each analyte of interest. Use the absolute retention time for each analyte as the midpoint of the window for that day. The daily retention time window equals the midpoint ± three times the standard deviation determined in Section 9.3.

9.5.4.1 Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window. Confirmation on a second GC column or by GC/MS analysis may be necessary.

9.5.4.2 Validation of GC system qualitative performance must be accomplished by the analysis of midlevel standards within the analysis sequence. If any of the standards fall outside their daily retention time window, the system is out of control. In such cases, the cause of the problem must be determined and corrected.

9.5.5 Aliphatic and aromatic ranges of interest are determined by the collective integration of all peak elutions between specified range “marker” compounds. Due to the variability in software approaches and applications to collective peak area integration, it is recommended that a manual check be initially performed, to document proper integration functions.

9.5.6 **When quantifying on a peak area basis by internal or external calibration, collective peak area integration for the fractional ranges, or TPH, must be from baseline (i.e. must include the unresolved complex mixture "hump" areas).** For the integration of individual Target Analytes, surrogate compounds, and internal standards, a valley-to-valley approach should typically be used, though this approach may be modified on a case-by-case basis by an experienced analyst.

9.5.7 Baseline correction using a system solvent blank is permissible, if conducted in accordance with the procedures and requirements specified in Section 11.2.5.

9.5.8 If the Target or Diesel PAH Analytes are to be quantitated using this method, and the response for an individual analyte exceeds the linear range of the system, dilute the extract and reanalyze. It is recommended that extracts be diluted so that all peaks are on scale. Overlapping peaks are not always evident when peaks are off scale. Computer reproduction of chromatograms, manipulated to ensure that all peaks are on scale over a 100-fold range, are acceptable if linearity is demonstrated prior to the analysis of samples. Peak height

measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.

9.5.9 For non-target analytes eluting in the aliphatic, aromatic or TPH fractions, the upper linear range of the system should be defined by peak height measurement, based upon the maximum peak height documented for an aliphatic or aromatic standard within the fraction that is shown to be within the linear range of the detector.

9.6 **Calculations**

9.6.1 External Standard Calibration

The concentration of each analyte and/or hydrocarbon range in a sample may be determined by calculating the amount of analyte or hydrocarbon range injected, from the peak response, using the calibration curve or the calibration factor determined in Section 9.4.

9.6.1.1 **Aqueous samples:**

The general equation to determine the concentration of a specific analyte or hydrocarbon range in aqueous samples in provided in Equation 7. If a Calibration Factor is used, the concentration of a specific analyte or hydrocarbon range may be calculated using Equations 8 and 9, respectively.

Equation 7

$$\text{Concentration } (\mu g/L) = \frac{(A_x) * (C_s) * (V_t) * (D)}{(A_s) * (V_s)}$$

where:

A_x = Response for the analyte, hydrocarbon range/TPH in the sample; units may be in area counts or peak height.

C_s = Concentration of the standard injected, (µg/mL).

A_s = Response for the external standard, units same as for A_x.

D = Dilution factor: if no dilution was made, D = 1, dimensionless.

V_t = Volume of total extract, mL. ***NOTE: This value will be 2.0-mL if 1-mL sample extract is diluted with 1-mL of the fractionation spiking solution.***

Vs = Volume of sample extracted, L.

Equation 8

$$\text{Concentration } (\mu g/L) = \frac{(A_x) * (V_t) * (D)}{(CF) * (V_s)}$$

where:

CF = Calibration Factor of individual analyte (area count/standard concentration).

Equation 9

$$(A_x) * (V_t) * (D)$$

$$\text{Concentration } (\mu g/L) = \frac{}{(\text{Range CF}) * (V_s)}$$

where:
Range CF = Calibration Factor for hydrocarbon range (collective area count/collective standard concentration).

9.6.1.2 **Nonaqueous samples:**
The general equation to determine the concentration of a specific analyte or hydrocarbon range in soil or sediment samples is provided in Equation 10. If a Calibration Factor is used, the concentration of a specific analyte or hydrocarbon range in a soil or sediment sample may be calculated using Equations 11 and 12, respectively..

Equation 10

$$\text{Concentration } (\mu g/kg) = \frac{(A_x) * (C_s) * (V_t) * (D)}{(A_s) * (W_d)}$$

where:
W_d = Dry weight of sample extracted, kg. (See Equations 15 through 17 for the calculation of the dry weight of soil and sediment samples)
A_x, C_s, A_s, V_t, and D, have the same definition as for aqueous samples.

Equation 11

$$\text{Concentration } (\mu g/kg) = \frac{(A_x) * (V_t) * (D)}{(CF) * (W_d)}$$

where:
CF = Calibration Factor of individual analyte (area count/standard concentration).

Equation 12

$$\text{Concentration } (\mu g/kg) = \frac{(A_x) * (V_t) * (D)}{(\text{Range CF}) * (W_d)}$$

where:
Range CF = Calibration Factor for hydrocarbon range (collective area count/collective standard concentration).

<u>9.6.2 Internal Standard Calibration</u>
The concentration of each analyte and hydrocarbon range in a sample may be determined by calculating the concentration of analyte or hydrocarbon range injected, from the peak response, based upon the analyte/internal-standard response ratio.

9.6.2.1 **Aqueous samples:**
The general equation to determine the concentration of a specific analyte or

hydrocarbon range in aqueous samples is provided in Equation 13.

Equation 13

$$\text{Concentration } (\mu g/L) = \frac{(A_x) * (C_{is}) * (V_t) * (D)}{(A_{is}) * (RF) * (V_s)}$$

where:
A_x = Response for the analyte, hydrocarbon range/TPH in the sample; units may be in area counts or peak height.
C_{is} = Concentration of the internal standard injected, (µg/mL).
A_{is} = Response of the internal standard, units same as A_x.
RF = Response factor for analyte or hydrocarbon range/TPH, dimensionless.
D = Dilution factor for instrumental analysis: if no dilution was made, D = 1, dimensionless.
V_t = Volume of total extract, mL. ***NOTE: This value will be 2.0-mL if 1-mL sample extract is diluted with 1-mL of the fractionation spiking solution.***
Vs = Volume of sample extracted, L.

9.6.2.2 **Nonaqueous samples:**
The general equation to determine the concentration of a specific analyte or hydrocarbon range in soil or sediment samples is provided in Equation 14.

Equation 14

$$\text{Concentration } (\mu g/kg) = \frac{(A_x) * (C_{is}) * (V_t) * (D)}{(A_{is}) * (RF) * (W_d)}$$

W_d = Dry weight of sample extracted, kg. (See Equations 15 through 17)
A_x, C_s, A_s, V_t, and D, have the same definition as for aqueous samples.

<u>9.6.3 Calculation of Dry Weight of Sample</u>
In order to calculate the dry weight of sample extracted (W_d), it is necessary to determine the moisture content of the soil/sediment sample, using the procedure outlined in Section 9.1.8. Using the data obtained from Section 9.1.8, W_d is calculated using Equations 15 through 17. The dry and wet weight for moisture determination are recorded in grams. The %Moisture and %DrySolids are recorded in percentages.

Equation 15

$$\%\ Moisture = \frac{(Wet\ sample\ weight - Dry\ sample\ weight)}{Wet\ sample\ weight} * 100$$

Equation 16

$$\%\ Dry\ Solids = (100) - (\%\ Moisture)$$

Equation 17

*W_d (kg) = (% Dry Solids/100) * (g of extracted sample/1000)*

10.0 QUALITY CONTROL

10.1 General Requirements and Recommendations

10.1.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of the data generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance standards for the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

10.1.2 A system solvent blank must be run after a sample suspected of being highly contaminated to determine if sample carryover has occurred.

10.1.3 At a minimum, for each analytical batch (up to 20 samples), a Calibration Check Standard (CCV), Laboratory Method Blank (LMB or MB), Laboratory Fortified Blank (LFB or LCS), and duplicate sample must be run. A Laboratory Fortified Matrix (LFM or MS) may be analyzed in duplicate (MSD), at the discretion of the analyst, based upon the nature of the sample. For analytical batches with more than 10 samples, the analysis of an additional mid-range calibration check standard should also be considered. The blank and spiked samples should be carried through all stages of the sample preparation and measurement process.

10.1.4 The recommended sequence of analysis is as follows:

(1) Calibration Standards (initial calibration or ICAL) or mid-range Calibration Check Standard (daily check of initial calibration or CCV) **[REQUIRED]**

(2) Laboratory Method Blank **[REQUIRED]**

(3) Laboratory Fortified Blank **[REQUIRED]**

(4) Samples

(5) Duplicate sample **[REQUIRED] – *NOTE: The analysts should employ MS duplicates instead of sample duplicates since the sample selected for duplicate analysis may not contain target analytes and precision information may be lost.***

(6) Laboratory Fortified Matrix/duplicate [As appropriate – See note above]

(7) Mid-range Calibration Check Standard [CCV, consider after 10 samples, as appropriate]

10.1.5 It is recommended that a system of control charts be developed to plot surrogate standard recoveries as a function of time. At a minimum, when surrogate recovery from a sample, blank, or QC sample is less than 40% or more than 140%, check calculations to locate possible errors, the fortifying solution for degradation, and changes in instrument performance. If the cause cannot be determined, reanalyze the sample.

10.2 Minimum Instrument QC

10.2.1 The instrument must be able to achieve adequate separation and resolution of peaks and analytes of interest.

> 10.2.1.1 The n-nonane (n-C9) peak must be adequately resolved from the solvent front of the chromatographic run.

10.2.1.2 The surrogates COD and OTP, fractionation surrogate standards, and any internal standards used must be adequately resolved from any individual components in the Aliphatic Hydrocarbon and Aromatic Hydrocarbon standards.

10.2.1.3 All peaks of interest from the Aliphatic Hydrocarbon standard must be adequately resolved to baseline. In the Aromatic Hydrocarbon standard, baseline separation is expected for Phenanthrene and Anthracene. The compounds, Benzo(a)Anthracene, Chrysene, Benzo(b)Fluoranthene, Benzo(k)flouranthene, Dibenzo(a,h)Anthracene, and Indeno(1,2,3-cd)Pyrene are not expected to be chromatographically separated to baseline; however, sufficient separation should be obtained to 50% baseline.

10.2.2 Retention time windows must be established for each analyte of interest each time a new GC column is installed, and must be verified and/or adjusted on a daily basis. (See Section 9.3).

10.2.3 Calibration curves must be developed based upon the analysis of calibration standards prepared at a minimum of 5 concentration levels. The linearity of calibration or response factors may be assumed if the percent relative standard deviation (%RSD) over the working range of the curve is less than or equal to 25%. Alternatively, if linear regression analysis is used for quantitation, the correlation coefficient (r) must be at least 0.99. (See Section 9.4)

10.2.4 The linear range of the aliphatic and aromatic hydrocarbon fractions, and/or TPH fraction, should be established based upon peak height. A series of analyses of high concentration Aliphatic and Aromatic Hydrocarbon Standards should be undertaken to establish the upper linear range of each component within the fractions of interest (< 25% RSD). The peak height associated with the highest concentration standard that is still within the linear range of response is then used to establish the upper limits of linearity for any individual non-target peak contained on a sample chromatogram within the range(s) of interest.

10.2.5 In order to demonstrate the absence of mass discrimination, the response ratio of C28 to C20 should be at least 0.85.

10.3 **Initial and Periodic Method QC Demonstrations**

The procedures specified in Section 10.3.1 through 10.3.3 must be conducted as an initial demonstration of laboratory capability, prior to the analysis of any samples. Subsequent to this initial demonstration, additional evaluations of this nature should be conducted on a periodic basis, in response to changes in instrumentation or operations, and/or in response to confirmed or suspected systems, method, or operational problems.

10.3.1 Accuracy and Precision

To demonstrate initial laboratory capability, analyze a minimum of four replicate reagent water and/or clean sand blanks spiked with each analyte of interest at approximately 50-µg/L and/or 5-mg/kg, respectively.

10.3.1.1 Extract and analyze each replicate according to the procedures described in Section 9.0.

10.3.1.2 Calculate the measured concentrations of each analyte in all replicates, the mean accuracy (as a percentage of true value) for each analyte, and the precision (as %RSD) of the measurements for each analyte.

10.3.1.3 For each analyte, excluding n-C36, the mean accuracy, expressed as a percentage of the true value, must be between 40% and 140%. Poorer recoveries may be

experienced for the n-C36 standard. For each analyte, the %RSD must be less than or equal to 25%.

10.3.1.4 If desired, the Accuracy and Precision evaluation may be combined with the MDL evaluation specified in Paragraph 10.3.2.

10.3.2 Method Detection Limits for Individual Analytes

Analyze a minimum of seven replicate reagent water and/or clean sand blanks which have been fortified with all analytes of interest at 3 to 5 times the calculated or estimated Instrument Detection Limits (IDLs). Typical spiking concentrations for analytes of interest are 5-µg/L for water, and 1.0-mg/kg for soil. Extract and analyze each replicate according to the procedures described in Section 9.0. Calculate the Method Detection Limit (MDL), Minimum Level (ML) or lowest level for reporting (LLR) and Reporting Limit (RL) or Method Quantitation Limit (MQL) of each analyte using the procedures described in Section 12.0.

10.3.2.1 Water MDLs are determined by extracting 7 to 10 replicates of 1-L reagent water blanks spiked with OTP, COD and each analyte of interest.

10.3.2.2 Soil/sediment MDLs are determined by extracting 7-10 replicates of 10-g of EPH-free sand blanks spiked with OTP, COD, and each analyte of interest.

10.3.2.3 For each analyte, excluding n-C36, the mean accuracy, expressed as a percentage of the true value, must be between 40% and 140%. Poorer recoveries may be experienced for the n-C36 standard. For each analyte, the %RSD must be less than or equal to 25%.

10.3.3 Method Detection Limits for Hydrocarbon Ranges

Analyze a minimum of seven replicate reagent water and/or clean sand blanks which have been fortified with diesel fuel at 3 to 5 times the calculated or estimated Instrument Detection Limit (IDL). Typical diesel fuel spiking concentrations are 0.1 mg/L for water, and 10 mg/kg for soil. Extract and analyze each replicate according to the procedures described in Section 9.0. Calculate the Method Detection Limit (MDL), Minimum Level (ML) or Lowest Level for Reporting (LLR) and Reporting Limit (RL) or Method Quantitation Limit (MQL) of each hydrocarbon range using the procedures described in Section 12.0.

10.3.3.1 Water MDLs are determined by extracting 7 to 10 replicates of 1-L reagent water blanks spiked with OTP, COD and the petroleum reference standard. Prepare a petroleum reference standard spiking solution by diluting the petroleum reference stock standard solution with acetone to a concentration of approximately 100-ng/µL. Spike a l-Liter reagent water blank with 1.0-mL of this spiking solution.

10.3.3.2 Soil/sediment MDLs are determined by extracting 7-10 replicates of 10-g sand blanks spiked with OTP, COD, and the petroleum reference standard. Prepare a petroleum reference standard spiking solution by diluting the petroleum reference stock standard solution with acetone to a concentration of approximately 100-ng/µL. Spike 10-g of clean sand blanks with 1.0-mL of this spiking solution.

10.3.3.3 The mean recovery of the summation of all (unadjusted) hydrocarbon ranges (i.e., including Target Analytes), expressed as a percentage of the true value of the petroleum reference standard, must be between 40% and 140%. For each hydrocarbon range, the %RSD must be less than or equal to 25%.

10.3.4 Fractionation

10.3.4.1 To demonstrate the capability of properly fractionating aliphatic and aromatic hydrocarbons in a sample, the analyst must first prepare and analyze the

Fractionation Check Solution specified in Section 7.7, using the fractionation procedures detailed in Section 9.1.4.

10.3.4.2 For each analyte within the Fractionation Check Solution, excluding n-C36, the mean accuracy, expressed as a percentage of the true value, must be between 40% and 140%

10.3.4.3 Subsequent to the initial demonstration of laboratory capability, it is recommended that a Fractionation Check Solution be analyzed for each new lot of silica gel/cartridges, to establish the optimum volume of hexane elution.

10.4 **Ongoing Method QC Demonstrations**

10.4.1 Each sample, blank, and Laboratory Fortified Blank must be spiked with the surrogate spiking solution. Required surrogate recovery is 40% to 140%. Recoveries outside this range must be noted and discussed on the data report form.

10.4.2 Each sample extract must be spiked with a fractionation surrogate spiking solution prior to fractionation. Required recovery is 40% to 140%. Recoveries outside this range must be noted and discussed on the data report form.

10.4.3 At a minimum, with every batch of 20 samples or less the lab must analyze the following:

10.4.3.1 **Calibration Check Standard (CCV)** - A mid-range calibration standard, prepared from the same stock standard solution used to develop the calibration curve, must be analyzed prior to sample analysis to verify the calibration state of the instrument. For large analytical batches that contain more than 10 samples, the analysis of an additional mid-range calibration check standard should also be considered after the analysis of the tenth sample. If the percent difference (%D) of any analyte within a calibration check standard varies from the predicted response by more than 25%, a new calibration curve must be prepared for that analyte. (See Section 9.4)

10.4.3.2 **Laboratory Method Blank (LMB or MB)** - A water or soil Laboratory Method Blank is prepared by fortifying a reagent water or clean sand blank with 1.0-mL of the surrogate spiking solution. Peaks detected within the retention time window of any analyte or range of interest above a Reporting Limit must be noted on the data report form.

10.4.3.3 **Laboratory Fortified Blank (LFB or LCS)** - A Laboratory Fortified Blank is prepared by fortifying a reagent water or clean sand blank with 1.0 mL of the matrix spiking solution. The spike recovery should be between 40% and 140%, excluding n-C36.

10.4.3.4 **Sample duplicate** - Sample duplicates may be laboratory duplicates, field duplicates, or matrix spike duplicates (MSD). The %RPD of duplicate samples must not exceed 50%.

10.4.3.5 **System Solvent Blank** - If baseline correction will be employed, as specified in Section 11.2.5, a system solvent blank, air blank, and/or system run must be undertaken with every batch, and after the analysis of a sample that is suspected to be highly contaminated. A system of control charts must be established to monitor instrument baseline as a function of time. In no case shall baseline correction be used if the instrument baseline is more than 25% greater than the average level established by these charts.

10.4.4 At the discretion of the analyst, and in consideration of sample matrices and data quality objectives, it is recommended that with every batch of 20 samples or less the lab consider

analysis of the following:

10.4.4.1 **Laboratory Fortified Matrix (LFM) /Duplicate** - The water or soil LFM is prepared by fortifying an actual water or soil sample with 1.0-mL of the matrix spiking solution. The purpose of the LFM spike is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations. The corrected concentrations of each analyte within the LFM spike sample should be within 40 to 140% of the true value, excluding n-C36. The %RPD of LFM Duplicates should be less than 50%.

10.4.5 If any of the performance standards specified in Section 10.4.1 through 10.4.3 are not met, the problem must be corrected before further samples are analyzed. Any samples run between the last QC samples that meet the criteria and those that are fallen out must be rerun. If this is not possible, that data must be reported as suspect.

10.4.6 The analyte and hydrocarbon range Reporting Limits should be verified/re-established at least once per year, or upon a major change in system equipment or operations.

11.0 DATA PRODUCTION AND REPORTING

11.1 **Calibration**

Using either the external calibration procedure (9.4.1) or the internal calibration procedure (9.4.2), calibrate the GC as follows:

11.1.1 Calculate an RF, CF or LR for the individual PAH compounds that comprise the Aromatic Hydrocarbon standard. This is not necessary if the Target or Diesel PAH Analytes will not be individually identified and quantitated using the EPH method.

11.1.3 Calculate an RF, CF or LR for the surrogates OTP and COD, and the fractionation surrogates.

11.1.4 Calculate a collective RF, CF or LR for the total mass concentration of the C9 -C18 Aliphatic Hydrocarbons. Tabulate the summation of the peak areas of all component standards in that fraction (e.g., C9-C18 Aliphatics, 6 components) against the total concentration of the standard that was injected.

11.1.5 Calculate a collective RF, CF or LR for the total mass concentration of the C19 -C36 Aliphatic Hydrocarbons. Tabulate the summation of the peak areas of all component standards in that fraction (e.g., C19-C36 Aliphatics, 8 components) against the total concentration of the standard that was injected. Do not include the surrogate COD.

11.1.6 Calculate a collective RF, CF or LR for the total mass concentration of the C11 -C22 Aromatic Hydrocarbons. Tabulate the summation of the peak areas of all component standards in that fraction (e.g., C11-C22 Aromatics, 17 components) against the total concentration of the standard injected. Do not include the surrogate OTP or the fractionation surrogate standard.

11.1.7 For TPH analyses, without fractionation, calculate a collective RF, CF, or LR. Tabulate the summation of the peak areas of all component standards in that fraction (i.e., 31 components) against the total concentration of the standard injected. Do not include any surrogates or internal standards.

11.2 **Sample Analysis**

11.2.1 <u>Aliphatic Fraction</u>

11.2.1.1 Determine the total area count for all peaks eluting 0.1 minutes before the

retention time (RT) for n-C9 and 0.01 minutes before the RT for n-C19. It is not necessary to identify or quantitate individual aliphatic compounds within this range.

11.2.1.2 Determine the total area count for all peaks eluting 0.01 minutes before the RT for n-C19 and 0.1 minutes after the RT for n-C36. It is not necessary to identify or quantitate individual aliphatic compounds within this range.

11.2.1.3 Determine the peak area count for the sample surrogate standard (COD) and any internal standard used. Subtract these values from the collective area count value within the appropriate hydrocarbon range(s).

11.2.1.4 Using the equations contained in Section 9.6 or linear regression analysis, calculate the collective concentrations of C9 through C18 Aliphatic Hydrocarbons, C19 through C36 Aliphatic Hydrocarbons, and individual concentrations of any sample surrogate and internal standards.

11.2.2 Aromatic Fraction

11.2.2.1 Determine the total area count for all peaks eluting 0.1 minutes before the retention time (RT) for naphthalene and 0.1 minutes after the RT for benzo(g,h,i)perylene.

11.2.2.2 Determine the peak area count for the sample surrogate (OTP), the fractionation surrogates, and any internal standard used. Subtract these values from the collective area count.

11.2.2.3 Optionally, determine the peak area count for the Target or Diesel PAH Analytes.

11.2.2.4 Using the equations contained in Section 9.6 or linear regression analysis, calculate the concentrations of Unadjusted C11 through C22 Aromatic Hydrocarbons, the sample surrogate standard (COD), the fractionation surrogates, and, optionally, the Target or Diesel PAH Analytes.

11.2.2.5 If the concentrations of the Target or Diesel PAH Analytes were determined, either by this method or another method, subtract the concentration of the Target or Diesel PAH Analytes from the concentration of Unadjusted C11 - C22 Aromatic Hydrocarbons. If the concentration of Target or Diesel PAH Analytes were not determined, report a value for unadjusted C11 - C22 Aromatics, and indicate "Not Determined" for C11 - C22 Aromatics.

11.2.3 Total Petroleum Hydrocarbons

11.2.3.1 Determine the total area count for all peaks eluting 0.1 minutes before the retention time (RT) for n-C9 and 0.1 minutes after the RT for n-C36. It is not necessary to identify or quantitate individual aliphatic compounds within this range.

11.2.3.2 Determine the peak area count for any surrogate and internal standards used. Subtract these values from the collective area count value.

11.2.3.3 Optionally, determine the peak area count for the Target or Diesel PAH Analytes.

11.2.3.4 Using the equations contained in Section 9.6 or linear regression analysis, calculate the concentration of Unadjusted TPH, and, optionally, the Target or Diesel PAH Analytes.

11.2.3.5 If the concentrations of the Target or Diesel PAH Analytes were determined, either by this method or another method, subtract the concentration of the Target or Diesel PAH Analytes from the concentration of unadjusted TPH. If the concentration of Target or Diesel PAH Analytes were not determined, report a value for Unadjusted TPH,

and indicate "Not Determined" for TPH.

11.2.4 Data Manipulations

11.2.4.1 By definition, the collective concentration of the aromatic fraction (and/or TPH) **excludes** the individual concentrations of the Target PAH Analytes.

Accordingly, a data manipulation step is necessary to adjust the collective range concentration calculated in 11.2.2.4 and 11.2.3.4, to eliminate "double counting" of analytes.

11.2.4.2 The necessary data manipulation step may be taken by the laboratory reporting the range/TPH concentration data, or by the data user. The extent of data manipulations taken by the laboratory must be noted on the data reporting form.

11.2.4.3 Subtract the individual concentrations of the Target or Diesel PAH Analytes from the collective concentration of Unadjusted C11 through C22 Aromatic Hydrocarbons and/or Unadjusted TPH. If the individual concentrations of Target Analytes have been quantitated using another method (e.g., by using an MS detector), note this on the data report form. If the individual concentrations of Target Analytes have not been quantitated, report a value for Unadjusted C11 through C22 Aromatic Hydrocarbons and/or Unadjusted TPH, and indicate "Not Determined" for C11 through C22 Aromatic Hydrocarbons and/or TPH.

11.2.4.4 For purposes of compliance with the reporting and cleanup standards specified in the Massachusetts Contingency Plan, the concentration of Unadjusted C11 through C22 Aromatic Hydrocarbons and/or Unadjusted TPH may be conservatively deemed to be equivalent to the concentration of C11 through C22

11.2.5 Baseline Correction for Instrument Noise Level

11.2.5.1 Range integration areas may be corrected by the manual or automatic subtraction of the baseline established by the injection of a system solvent blank. Correction in this manner is not recommended or preferred, but is permissible in cases where all reasonable steps have been taken to eliminate or minimize baseline noise and rise.

11.2.5.2 Instrument baseline shall be established by the direct injection of a system solvent blank. Alternatively, this level may be established by the injection of an air blank, or by activation of a programmed run without the injection of any material. All system and run elements and parameters must be identical to those of the sample run.

11.2.5.3 If baseline correction is used, instrument baseline must be established for every batch of samples, and after the analysis of samples that are suspected to be highly contaminated. Baseline correction can not be used on any sample in which the instrument baseline level is 25% above mean response levels, as established by a system of control charts that must be established to monitor instrument baseline as a function of time.

11.2.6 Blank Correction for SPE Cartridges

11.2.6.1 Range integration areas may be corrected by the manual or automatic subtraction of peaks identified during the injection of a Laboratory Method Blank, and determined to be attributable to the leaching of plasticizers or other contaminants from silica gel SPE cartridges. Correction in this manner is not recommended or preferred, but is permissible in cases where all reasonable steps have been taken to eliminate or minimize system contamination problems arising from the use of such products.

11.2.6.2 A blank correction undertaken in this manner constitutes a significant modification of the method, and must be noted on the data report form.

11.3 **Data Reporting Format**

11.3.1 The required data reporting format is presented in Appendices 3 and 4. The purpose of this format is to provide data users with a succinct and complete summary of pertinent information and data, a clear indication of whether significant modifications were made to the EPH Method, and a clear affirmation on whether the QA/QC procedures and standards specified in this method where followed and achieved. While it is permissible to alter the form and presentation of data, all of the information must be provided in a clear, concise, and succinct manner.

11.3.1.1 "Significant Modifications" to this method shall include, without limitation, all of the following:

11.3.1.1.1 The use of other than a silica-gel fractionation technique;

11.3.1.1.2 The use of a soil extraction procedure other than Soxhlet, Soxtec, or sonication;

11.3.1.1.3 The use of solvents other than those recommended in this method;

11.3.1.1.4 The use of a detector other than a Flame Ionization Detector (FID) to quantitate range/TPH concentrations;

11.3.1.1.5 The use of surrogate compounds with relative retention times less than the recommended compounds;

11.3.1.1.6 The use of a fractionation surrogate compound with a relative retention time greater than the recommended compounds;

11.3.1.1.7 The use of blank corrections per 11.2.6; or

11.3.1.1.8 Failure to provide all of the information and data required in the reporting format presented in Appendices 3 and 4.

11.3.1.2 Positive affirmation that all required QA/QC procedures and performance standards were followed and achieved means that all of the required steps and procedures detailed in Section 10.0 have been followed, and that all data obtained from these steps and procedures were within the acceptance limits specified for these steps and procedures.

12.0 **REPORTING LIMITS**

The Reporting Limits or Method Quantitation Limits (RLs or MQLs) for individual analytes and hydrocarbon ranges shall be experimentally determined by each laboratory using this method. Although laboratories have flexibility in performing this task, in order to ensure the validity and meaningfulness of these concentration values, the following performance standards must always be achieved:

- The RLs (MQLs) for individual analytes must be established by the analyses of at least 7 replicate water and/or sand blanks fortified with the individual analytes of interest;

- The RLs (MQLs) for the hydrocarbon ranges must be established by the analyses of at least 7 replicate water and/or sand blanks fortified with a petroleum reference standard;

- The RL (MQLs) for individual analytes may be set no higher than the lowest calibration standard which is still within the linear range of the calibration curve (i.e, less than 25% RSD), assuming 100% extraction of the sample matrix; and

- The RL (MQLs) for individual analytes must be verified by the analyses of at least 4 replicate water or sand blanks fortified at the Reporting Limit, where the precision of replicate analyses is demonstrated to be equal to or less than 25% RSD, and the mean accuracy is demonstrated to be between 40-140% of the spiked value.

12.1 **Individual Analytes**
The Reporting Limit or Method Quantitation Limit (RL or MQL) for individual analytes of interest should be calculated by first determining a Method Detection Limit (MDL) for each analyte, and then applying an adjustment factor to the MDL to calculate the Minimum Level (ML).
12.1.1 The MDL is determined by the analysis of 7-10 replicate samples, as detailed in Section 10.3.2. The data obtained from these analyses are then use to calculate the MDL using Equation 18. Student t values are shown in Table 7.

Equation 18

$MDL = (t) * (SD)$

where:
t = student t value at the 99% confidence level.
SD = standard deviation of the replicate analysis.

12.1.2 According to the MADEP-EPH method, the Minimum Level (ML) is then calculated from the MDL according to equation 19:

Equation 19

$ML = (MDL) * 3.18$

12.1.3 According to the MADEP-EPH Method, if the ML is greater than the concentration of analyte used in the MDL study, the Reporting Limit is this value. If the ML is less than the concentration of analyte used in the MDL study, the laboratory may either (a) establish the RL at the concentration of the analyte used in the MDL study, or (b) undertake additional analyses to demonstrate method performance at the ML.
12.1.3.1 To demonstrate adequate method performance at the calculated ML and proposed RL, analyze at least 4 replicate water and/or sand samples fortified with the analytes of interest at the ML, according to the MADEP-EPH Method.
12.1.3.2 The mean recovery of each analyte, expressed as a percentage of the true value, must be between 40-140%. For each analyte, the %RSD of replicate analyses must be less than or equal to 25%, according to the MADEP-EPH Method.
12.1.4 According to the MADEP-EPH Method, a low-level calibration standard must be prepared, if necessary at the concentration of the proposed RL, assuming 100% extraction from the sample matrix. This standard must be analyzed and shown to be within the linear range of the calibration curve (i.e., %RSD equal to or less than 25%). Note that the recommended analyte concentrations for the MDL studies specified in Section 13.3.2 are equivalent to the lowest calibration standard recommended in Table 5, assuming 100% extraction from the sample, and

the recommended sample preparation and analysis procedures specified in this method.
12.1.5 AML will employ the following pragmatic approach for MQL based on the rationale described below. The concentration of the lowest point on the initial calibration curve is determined based on information available from closely related methods (e.g. EPA 610, SW-846 8100, SW-846 8015, etc.), the sensitivity of the GC/FID detector and from the experience of the analyst. The difficulty in preparing custom calibration solutions with different concentrations for different compounds is also a big deterrent in employing the ML concentration to define the reporting limit. At AML, the lowest point on the initial calibration curve, which will serve to define the method quantitation limit will be selected at levels that are slightly greater than ML.

12.2 **Collective Hydrocarbon Ranges/TPH**
According to the MADEP-EPH Method, the Reporting Limit (RL) for each hydrocarbon range, and/or for TPH should be calculated by first determining a Method Detection Limit (MDL) for each range and/or for TPH, and then applying an adjustment factor to the MDL to calculate the Minimum Level (ML).
12.1.1 The MDL is determined by the analysis of 7-10 replicate samples, as detailed in Section 10.3.3. The data obtained from these analyses are then used to calculate the MDL using Equation 18.
12.1.2 The Minimum Level (ML) is then calculated from the MDL according to equation 19.
12.1.3 If the ML is greater than the mean concentration of the hydrocarbon range determined in the MDL study, the Reporting Limit is this value. If the ML is less than the mean concentration of the hydrocarbon range determined in the MDL study, the laboratory may either (a) establish the RL at the concentration of the hydrocarbon range
Extractable Petroleum Hydrocarbons determined in the MDL study, or (b) undertake additional analyses to demonstrate method performance at the ML.

12.1.3.1 To demonstrate adequate method performance at the calculated ML and proposed RL, analyze at least 4 replicate water and/or sand samples fortified with the petroleum reference standard at or near the range MLs of interest. For range MLs, this will involve assumptions on the chemistry of the petroleum reference standard, based upon data obtained during the MDL studies.
12.1.3.2 The mean recovery of the summation of each range, and/or TPH, expressed as a percentage of the true value of the petroleum reference standard, must be between 40-140%. For each range and/or TPH, the %RSD of replicate analyses must be less than or equal to 25%.
12.1.3.3 AML will employ the following pragmatic approach for MQL based on the rationale described below. The concentration of the lowest point on the initial calibration curve is determined based on information available from closely related methods (e.g. EPA 610, SW-846 8100, SW-846 8015, etc.), the sensitivity of the GC/FID detector and from the experience of the analyst. The difficulty in preparing custom calibration solutions with different concentrations for different compounds is also a big deterrent in employing the ML concentration to define the reporting limit. At AML, the lowest point on the initial calibration curve, which will serve to define the method quantitation limit will be selected at levels that are slightly greater than ML.

13.0 METHOD PERFORMANCE

Single laboratory accuracy, precision and MDL data for method analytes are provided in Tables 1-1 through 1-4 in Appendix 1. Chromatograms are provided in Appendix 2.

14.0 REFERENCES

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14.2 USEPA, "Measurement of Petroleum Hydrocarbons: Report on Activities to Develop a Manual" Prepared by Midwest Research Institute, Falls Church, VA, under EPA Contract #68-WO-0015, WA No. 4; submitted to USEPA Office of Underground Storage Tanks, Washington, DC; November 20, 1990.

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Determination of Extractable Petroleum Hydrocarbons (EPH) – MADEP-EPH-98-1 for the Massachusetts Contingency Plan (MCP), BWSC-CAM, Interim Final, July 2002.

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14.19 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

14.20 14.16 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.

14.21 14.17 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

14.22 AML Standard Operating Procedures, Q-Sonicator-RD.

14.23 AML Standard Operating Procedures, Q-Water-RC.

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14.27 Determination of total purgeable (diesel range) petroleum hydrocarbons (OA-2), State of Iowa, 1991.

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14.29 NWTPH-Dx - Semivolatile Petroleum Products Method for Soil and Water, Northwest (Oregon and Washington) Total Petroleum Hydrocarbon Analytical Methods, State of Oregon DEQ lab, December 1996.

14.30 NWTPH-HCID - Hydrocarbon Identification Method for Soil and Water, Northwest (Oregon and Washington) Total Petroleum Hydrocarbon Analytical Methods, State of Oregon DEQ lab, December 1996.

14.31 Petroleum Range Organics, FL-PRO, State of Florida.

Table 1. Method Performance

Matrix	# Labs Proficient	% Labs Proficient	Data from Proficient Laboratories		
			Fraction	%RSD	% Labs within ±40% mean value
Soil	19	83	C9-C18 Aliphatics	23	95
			C19-C36 Aliphatics	30	89
			C11-C22 Aromatics	19	100
			Total All Fractions (TPH)	17	100
Water	20	87	C9-C18 Aliphatics	84	22
			C19-C36 Aliphatics	192	94
			C11-C22 Aromatics	47	72
			Total All Fractions (TPH)	35	83

Table 2. Aliphatic Hydrocarbon Standard

Carbon Number	Compound	Retention Time (min.)*
9	n-Nonane	3.14
10	n-Decane	4.55
12	n-Dodecane	7.86
14	n-Tetradecane	11.10
16	n-Hexadecane	14.05
18	n-Octadecane	16.71
19	n-Nonadecane	17.95
20	n-Eicosane	19.14
	1-Chloro-octadecane (surrogate)	20.13
22	n-Docosane	21.35
24	n-Tetracosane	23.40
26	n-Hexacosane	25.29
28	n-Octacosane	27.04
30	n-Triacontane	28.69
36	n-Hexatriacontane	34.82

*Results obtained using the column and chromatographic conditions described in Sections 6.4 and 9.2, respectively of this SOP document.

Table 3. Aromatic Hydrocarbon Standard/Target PAH Analytes

Compound	Retention Time (min.)*	MDL** Water (µg/kg)	MDL** Soil (mg/kg)
Naphthalene	7.23	0.14	0.09
2-Methylnaphthalene	9.58	0.18	0.09
Acenaphthylene	12.28	0.14	0.09
Acenaphthene	12.84	0.16	0.09
Fluorene	14.45	0.25	0.09
Phenanthrene	17.28	0.31	0.16
Anthracene	17.44	0.30	0.16
Ortho-Terphenyl (surrogate)	…….	0.31	0.13
Fluoranthene	20.85	0.47	0.16
Pyrene	21.45	0.47	0.16
Benzo(a)Anthracene	25.16	0.60	0.19
Chrysene	25.25	0.60	0.19
Benzo(b)Fluoranthene	28.18	0.60	0.19
Benzo(k)Fluoranthene	28.26	0.66	0.16
Benzo(a)Pyrene	28.97	0.50	0.16
Indeno(1,2,3-cd)Pyrene	31.63	0.41	0.13
Dibenzo(a,h)Anthracene	31.75	0.44	0.13
Benzo(g,h,i)Perylene	32.15	0.57	0.13

*Results obtained using the column and chromatographic conditions described in Sections 6.4 and 9.2, respectively of this SOP document.
**Single laboratory MDL study (reproduced from MADEP method).

Table 4. Holding Times and Preservatives for EPH Samples

Matrix	Container	Preservation	Holding Time
Aqueous Samples	1-Liter amber glass bottle with Teflon-lined screw cap. Minimum = 1; Maximum = 2 per sample .	Add 5-mL of 1:1 HCl; cool to 4 °C.	Extract within 14-days of collection. Analyze within 40-days of extraction.
Soil/ Sediment Samples	One 4-Oz. (120-mL) wide-mouth amber glass jar with Teflon-lined screw cap.	Cool to 4 °C.	Extract within 7-days of collection. Analyze within 40-days of extraction.

Table 5. EPH Marker Compounds

Range/ Hydrocarbon Standard	Beginning Marker Compound	Ending Marker Compound
C9-C18 Aliphatic Hydrocarbons	Just (0.01-min.) before n-Nonane	Just (0.01-min.) before n-Nonadecane
C19-C36 Aliphatic Hydrocarbons	Just (0.01-min.) before n-Nonadecane	Just (0.01-min.) after n-Hexatriacontane
C11-C22 Aromatic Hydrocarbons	Just (0.01-min.) before Naphthalene	Just (0.01-min.) after Benzo (g,h,i) Perylene

Table 6. Recommended Calibration Standard Concentrations

Component	**Concentration of standard analytes (ng/µL or µg/mL)**						
	10	**20**	**50**	**70**	**100**	**150**	**200**
Total Concentration of C9 - C18 Aliphatic Hydrocarbons (**µg/mL)** (6 components)	60	120	300	420	600	900	1200
Total Concentration of C19 - C36 Aliphatic Hydrocarbons (**µg/mL)** (8 components)	80	160	400	560	800	1200	1600
Total Concentration of C11 - C22 Aromatic Hydrocarbons / PAHs (**µg/mL)** (17 components)	170	340	850	1190	1700	2550	3400

Table 7. Student t values for MDL Calculation

Number of Replicates	T value for 99% confidence interval
7	3.14
8	3.00
9	2.90
10	2.82

STANDARD OPERATING PROCEDURES

No. O-HAA-RA

Haloacetic Acids by Microextraction, Derivatization and GC/ECD Analysis (EPA Methods 552.1, 552.2, and 552.3)

January 17, 2006

APPROVED:

_____ _____
Laboratory Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Haloacetic Acids by Microextraction, Derivatiztion, and GC/ECD Analysis (EPA Methods 552.1, 552.2, and 552.3)

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) based on EPA methods 552.1, 552.2, and 552.3 is used to determine the concentrations of the following compounds in water samples.

Compound	CAS Number
Bromochloroacetic Acid (BCAA)	5589-96-3
Bromodichloroacetic Acid (BDCAA)	7113-314-7
Chlorodibromoacetic Acid (CDBAA)	5278-95-5
Dalapon	75-99-0
Dibromoacetic Acid (DBAA)	631-64-1
Dichloroacetic Acid (DCAA)	79-43-6
Monobromoacetic Acid (MBAA)	79-08-3
Monochloroacetic Acid (MCAA)	79-11-8
Tribromoacetic Acid (TBAA)	75-96-7
Trichloroacetic Acid (TCAA)	76-03-9

1.2 This SOP employs microextraction and analysis by gas chromatography using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD).

1.3 The experimentally determined method detection limits (MDL) varies with the compound and they are generally < 1 µg/L. The method has been shown to be useful for these analytes over a concentration range of approximately 1 to 50 µg/L. Actual detection limits are highly dependent upon the characteristics of the gas chromatographic system, sample matrix, and calibration. See Table 1 for MDLs obtained at the laboratory.

1.4 This EPA procedure employs a Procedural Standard Calibration, which is a calibration method where aqueous calibration standards are prepared and processed (e.g., purged, extracted and/or derivatized) in exactly the same manner as a sample. All steps in the process from addition of sampling preservatives through instrumental analyses are included in the calibration. Using procedural standard calibration compensates for any inefficiencies in the processing procedure.

1.5 This method is restricted to use by or under the Supervision of analysts experienced in the use of gas chromatography and in the interpretation of gas chromatograms. Each analyst should demonstrate the ability to generate acceptable results with this method.

1.6 The toxicity of the target compounds is not been well established. Pure standard materials and stock standard solutions of these compounds should be handled in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these compounds.

2.0 SUMMARY OF METHOD

2.1 A 40 mL volume of sample is adjusted to pH <0.5 and extracted with 4 mL of methyl-tert-butyl-ether (MTBE). The haloacetic acids that have been partitioned into the organic phase are then converted to their methyl esters by the addition of acidic methanol followed by slight heating. The acidic extract is neutralized by a backextraction with a saturated solution of sodium bicarbonate and the target analytes are identified and measured by capillary column gas chromatography using an electron capture detector (GC/ECD). Analytes are quantitated using procedural standard calibration.

2.2 Calibration standards, laboratory control samples (LCS) and aqueous matrix spikes (MS) are extracted and analyzed in an identical manner as the samples in order to compensate for possible extraction losses.

2.3 The extraction and analysis time is 30 to 50 minutes per sample depending upon the analytical conditions chosen.

2.4 Confirmatory evidence is obtained using a different column.

3.0 INTERFERENCES

3.1 Impurities contained in the extracting solvent (MTBE) usually account for the majority of the analytical problems. Reagent blanks should be analyzed for each new bottle of hexane before use. Indirect daily checks on the hexane are obtained by monitoring the reagent blanks. Whenever an interference is noted in the method or instrument blank, the laboratory should reanalyze the MTBE and obtain a new source of MTBE solvent.

3.2 Bromochloroacetic acid coelutes with an interferant on the DB-1701 confirmation column. The interferant has been tentatively identified as dimethyl sulfide. However, because of the difference in peak shapes, the peak for the ester of BCAA tends to "ride on" the interferant peak and quantitative confirmation can be performed by manual integration that includes only the peak area of the target ester.

3.3 Methylation using acidic methanol results in a partial decarboxylation of tribromoacetic acid . Therefore a substantial peak for bromoform will be observed in the chromatograms. Its elution does not, however, interfere with any other analytes. Furthermore, this demonstrates the need for procedural standards to establish the calibration curve by which unknown samples are quantitated.

3.4 This liquid/liquid extraction technique extracts a wide boiling range of non-polar organic compounds and, in addition, extracts some polar organic compounds. The extent of matrix interferences will vary considerably from source to source, depending upon the water sampled. Analyte identifications should be confirmed using the confirmation column or by GC/MS if the concentrations are sufficient.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph - A Hewlett Packard 5890 Series II GC is used. The system is complete with a temperature-programmable GC for splitless injection. The system includes all accessories, including syringes, analytical columns and gasses. The column flow rate remains constant throughout temperature program operation. The capillary column is directly coupled to the source.

4.2 Gas chromatographic columns

4.2.1 Primary GC Column -- DB-5.625 [fused silica capillary with chemically bonded (5% phenyl)-methylpolysiloxane)] or equivalent bonded, fused silica column, 30m x 0.25mm ID, 0.25 µm film thickness.

4.2.2 Confirmation GC Column -- DB-1701 [fused silica capillary with chemically bonded (14% cyanopropylphenyl)-methylpolysiloxane)] or equivalent bonded, fused silica column, 30 m x 0.25 mm ID, 0.25 µm film thickness.

4.3 Data system - Hewlett Packard's Environquant is used to allow continuous acquisition and storage on machine-readable media of all data obtained throughout the duration of the GC program.

4.4 Microsyringes - 10, 25, 100, 250, 500 and 1000 uLs.

4.5 Balance - Analytical, 0.0001 grams and top loading, 0.1 grams.

4.6 Disposable pipettes – Pasteur

4.7 Graduated Conical Centrifuge Tubes with Teflon-Lined Screw Caps - 15 mL with graduated 1 mL markings.

4.8 Volumetric flasks, Class A – 5 mL and 10 mL.

4.9 Glass bottles – 10 to 20 mL, with Teflon lined screw caps or crimp tops.

4.10 Graduated cylinder - 50 mL.

4.11 Transfer pipet.

4.12 Sample Containers - Amber glass bottles, approximately 40 to 50 mL, fitted with Teflon-lined screw caps.

4.13 Extraction Vials - 60 mL clear glass vials with teflon-lined screw caps.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Organic-free reagent water - Use Type 1 water (see applicable AML SOP on reagent water).

5.3 Methyl-tert-Butyl Ether, High Purity -- Demonstrated to be free of analytes and interferences, redistilled in glass if necessary.

5.4 Methyl alcohol, CH_3OH - Demonstrated to be free of analytes.

5.5 Sodium chloride, NaCl - Pulverize a batch of NaCl and place it in a muffle furnace at room temperature. Increase the temperature to 400°C for 30 minutes. Store in a capped bottle.

5.6 Sodium Sulfate, Na_2SO_4, ACS Granular, Anhydrous -- If interferences are observed, it may be necessary to heat the sodium sulfate in a shallow tray at 400°C for up to four hours to remove phthalates and other interfering organic substances. Alternatively, it can be extracted with methylene chloride in a Soxhlet apparatus for 48 hours. Store in a capped glass bottle rather than a plastic container.

5.7 Copper II Sulfate Pentahydrate, CuSO 5H O, ACS Reagent Grade.

5.8 Sodium Bicarbonate, NaHCO , ACS Reagent Grade.

5.9 Ammonium Chloride, NH Cl, ACS Reagent Grade - Used to convert free chlorine to monochloramine. Although this is not the traditional dechlorination mechanism, ammonium chloride is categorized as a dechlorinating agent in this method.

5.10 Solutions

5.10.1 10% H2SO4 / methanol Solution -- Use caution when preparing sulfuric acid solutions. To prepare a 10% solution, add 5 mL sulfuric acid dropwise (due to heat evolution) to 20-30 mL methanol contained in a 50.0 mL volumetric flask that has been placed in a cooling bath. Then dilute to the 50.0 mL mark with methanol.

5.10.2 Saturated Sodium Bicarbonate Solution -- Add sodium bicarbonate to a volume of water, mixing periodically until the solution has reached saturation.

5.11 Standards

5.11.1 7.5.1 1,2,3-trichloropropane, 99+% - For use as the internal standard. Prepare an internal standard stock standard solution of 1,2,3-trichloropropane in MTBE at a concentration of approximately 1 mg/mL. Alternatively, purchase a solution of it from Ultra Scientific (Cat # PPS-251) at a concentration of 1000 µg/mL in MTBE. From this stock standard solution, prepare a primary dilution standard in MTBE at a concentration of 25 µg/mL.

5.11.2 2,3-dibromopropionic Acid, 99+% - For use as a surrogate compound. Prepare a surrogate stock standard solution of 2,3-dibromopropionic acid in MTBE at a concentration of approximately 1 mg/mL. Alternatively, purchase a solution of it from Ultra Scientific (Cat # PPS-390) at a concentration of 1000 µg/mL in MTBE. From this stock standard solution, prepare a primary dilution standard in MTBE at a concentration of 10 µg/mL.

5.12 Stock standards - These solutions may be purchased as certified solutions from Ultra Scientific (Cat # PHM-5524A0 at a concentration of 0.2 to 1 mg/mL) and Supelco (Cat # 47787 for second-source) in MTBE. ***Note:*** When purchasing commercially prepared standards, solutions prepared in methanol must not be used because it has been found that the haloacetic acids are subject to spontaneous methylation when stored in this solvent . Furthermore, tribromoacetic acid has been found to be unstable in methanol because it undergoes decarboxylation when stored in this solvent. Alternatively the stock standard mixture may be prepared from pure standards using the following procedures:

5.12.1 For analytes which are solids in their pure form, prepare stock standard solutions by accurately weighing approximately 0.01-0.05 g of pure

material in a 10.0 mL volumetric flask. Dilute to volume with MTBE. When a compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard.

5.12.2 Stock standard solutions for analytes which are liquid in their pure form at room temperature can be accurately prepared in the following manner.

5.12.2.1 Place about 9.8 mL of MTBE into a 10.0 mL volumetric flask. Allow the flask to stand, unstoppered, for about 10 minutes to allow solvent film to evaporate from the inner walls of the volumetric, and weigh to the nearest 0.1 mg.

5.12.2.2 Use a 10 µL syringe and immediately add 10.0 µL of standard material to the flask by keeping the syringe needle just above the surface of the MTBE. Be sure that the standard material falls dropwise directly into the MTBE without contacting the inner wall of the volumetric.

5.12.2.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in milligrams per milliliter from the net gain in weight.

5.12.3 Purchase a second source standard mixture from Supelco (Cat # 47784) at a concentration of 0.2 to 1 mg/mL.

5.12.4 Store stock standards in amber vials equipped with Teflon lined screw-caps or crimp tops. Stock standards are stable for at least four weeks when stored at 4°C and away from light.

5.13 Primary Dilution Standard (PDS) - Use stock standards to prepare the PDS intermediate standard that contains the analytes in MTBE. The intermediate standard should be prepared at a concentration that can be easily diluted to prepare aqueous calibration standards that will bracket the working concentration range. Prepare the primary dilution standard solution by combining and diluting stock standard solutions with MTBE and the surrogate stock standard solution. This primary dilution standard solution should be stored at -10°C and protected from light. It is stable for at least one month but should be checked before use for signs of evaporation. As a guideline to the analyst, use the primary dilution standard solution that was used in the validation of this method by EPA.

	Concentration, µg/mL
Monochloroacetic Acid	60
Monobromoacetic Acid	40
Dalapon	40
Dichloroacetic Acid	60
Trichloroacetic Acid	20
Bromochloroacetic Acid	40
Dibromoacetic Acid	20
Bromodichloroacetic Acid	40
Chlorodibromoacetic Acid	100
Tribromoacetic Acid	200
2,3-Dibromopropionic Acid (Surrogate)	100

Include the surrogate analyte, 2,3-dibromopropionic acid, within the primary dilution standard. By incorporating the surrogate into the primary dilution standard, it is diluted alongside the target analytes in the standard calibration curve. This is done so that the peaks for the surrogate and the ester of chlorodibromo-acetic acid, which elute fairly closely, are relatively close in size and adequate resolution is therefore insured. Furthermore, if a sample should have a very large concentration of chlorodibromo-acetic acid, it may be impossible to obtain an accurate measurement of surrogate recovery. If this happens, reextraction with a higher surrogate concentration would be an option. This primary dilution standard is used to prepare calibration standards, which comprise five concentration levels of each analyte with the lowest standard being at or near the MDL of each analyte. The concentrations of the other standards should define a range containing the expected sample concentrations or the working range of the detector.

5.14 Initial Calibration Standards - A minimum of 5 initial calibration solutions (ICAL) are prepared. Various volumes of the PDS solution is diluted in 40-mL (final volume) of reagent water to prepare the ICAL standards. Measure out 40-mL of reagent water into 40-mL VOA vials. Remove volumes of water that equal the volume of the PDS solution that will be spiked into them. Immediately after spiking the PDS solution, repace the cap and swirl the container for uniform mixing.

5.15 Initial Calibration verification (ICV) or Quality control reference (QC) standards - Prepare a mid-point initial calibration verification standard using a second source PDS.

5.16 Continuing Calibration Verification Standard (CCV) - Prepare several CCV solutions at the mid-point of the ICAL using PDS from the same source as the ICAL standards. Prepare at the same levels as the ICV.

5.17 Method Blank - Prepare method blank (MB) using the same procedure as ICAL standards. The spike volume is zero

5.18 Laboratory Control Samples - Prepare exactly as described for CCV. Prepare in duplicate (LCS and LCSD).

5.19 Matrix Spike Samples - Prepare in duplicate. Measure out 40-mL of the sample into 60-mL VOA vials. Remove volumes of water that equal the volume of the PDS solution that will be spiked into them. Immediately after spiking the PDS solution, repace the cap and swirl the container for uniform mixing. The spiked MS and MSD samples are now ready for analysis.

5.19.1 Surrogate - The surrogate is included in the PDS solution that is used to prepare the spiked samples (LCS/LCSD, MS/MSD) and the calibration verification (ICV/CCV) standards. Us the surrogate level in these samples/standards for spiking environmental samples.

6.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

6.1 Groundwater, Surface water, and Wastewater samples - Amber 60-mL glass screw-cap VOA vials with Teflon lined silicone septa may be used for collecting these aqueous samples. Order pre-cleaned 60-mL VOA vials not containing the hydrochloric acid preservative directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 2 vials for each water sample.

6.1.1 Groundwater, surface water and wastewater samples that have not been chlorinated do not require the addition of ammonium chloride. IF they are chlorinated, use procedures described for the drinking water.

6.1.2 Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. Provide two sets of 2 vials for the MS and MSD samples.

6.1.3 When sampling from a well, fill a wide-mouth bottle or beaker with sample, and carefully fill the 60-mL sample bottles.

6.2 Soil samples collected with little headspace in 2-Oz or 4-Oz jars do not require chemical preservation.

6.3 Drinking water samples - The procedure described for water sampls (Section 6.1) is used with the following modifications.

6.3.1 The samples are preserved with ammonium chloride for drinking water samples to convert the free residual chlorine in the sample matrix to combined chlorine. Prior to shipment to the field, add crystalline or granular ammonium chloride (NH_4Cl) to the sample container in an amount to produce a concentration of 100 mg/L in the sample. For a typical 60 mL sample, 6 mg of ammonium chloride is added. ***Note***: Enough ammonium chloride must be added to the sample to convert the free chlorine residual in the sample matrix to combined chlorine. Typically, the ammonium chloride concentration here will accomplish that. If high doses of chlorine are used, additional ammonium chloride may be required.

6.3.2 When sampling from a tap, open the tap and allow the system to flush until the water temperature has stabilized (usually about 10 minutes). Adjust the flow to about 500-mL/min and collect the sample from the flowing stream. After collecting the sample in the bottle containing the ammonium chloride, seal the bottle and agitate by hand for one minute.

6.4 The samples must be chilled to 4°C or less at the time of collection and maintained at that temperature during shipment. Sample should be packaged with sufficient ice during shipment for the samples to be maintained at 4°C or less on arrival at the laboratory. Upon receipt and log-in, samples should be stored in the walk-in cooler at 4°C.

6.4.1 Samples preserved at 4°C with chemical preservatives, when required must be extracted within the specified holding times (14-days) for the results to be considered reflective of total concentrations. Sample extracts must be analyzed within 7 days from extraction if stored at 4°C or within 14 days if stored at -10°C or less.

6.4.2 Analytical data generated outside of the specified holding times should be considered to be minimum values only. Such data may be used to

demonstrate that the concentration of a constituent to be above the regulatory threshold but cannot be used to demonstrate for compliance with regulatory limits.

7.0 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control contain a number of regulated compounds at high concentrations. The toxicity of these compounds present in such complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 The toxicity of the extraction solvent, MTBE, has not been well defined. Susceptible individuals may experience adverse affects upon skin contact or inhalation of vapors. Therefore protective clothing and gloves should be used and MTBE should be used only in a chemical fume hood or glove box. The same precaution applies to pure standard materials.

7.5 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Aqueous and soil samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters after the solvent(s) have been allowed to evaporate in a hood.

9.0 PROCEDURE

9.1 Gas Chromatographic Conditions - Two gas chromatography columns (DB-5 and DB-1701) are recommended in the referenced EPA methods. Column 1 can be used as the primary analytical column. Column 2 is recommended in the referenced methods for use as a confirmatory column. Splitless injection with 30 second dealy is recommended with the injected sample split for the dual column analysis. Although the following temperature program is recommended, the analyst may use other programs that provide adequate separation of the target compounds.

9.1.1 Primary and Confirmation Columns:

Injector temperature:	200°C.
Detector temperature:	260°C.
Carrier gas (Helium)Linear velocity:	25 cm/sec.

Temperature program:

Initial temperature:	40°C, hold for 10 min.
Program Ramp-1:	40°C to 75°C at 5°C/min.
Hold for Ramp-1:	15 minutes @ 75°C
Program Ramp-2:	75°C to 100°C at 5°C/min.
Hold for Ramp-2:	5 minutes @ 100°C
Program Ramp-3:	100°C to 135°C at 5°C/min.
Hold for Ramp-3:	2 minutes @ 135°C

9.2 Calibration

9.2.1 Prepare at least five initial calibration standards (ICAL) as described in Section 5.10. The lowest ICAL standrad should contain the target compounds at a concentration near, but greater than, the method detection limit for each compound. The others should be at concentrations that bracket the range expected in the samples.

9.2.2 Perform micro-extraction on ICAL standards and analyze the MTBE extracts from each calibration standard. Tabulate peak height or area response versus the concentration in the standard. Prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (< 20% relative standard deviation), linearity can be assumed and the average ratio or calibration factor can be used in place of a calibration curve. Refer to other appropriate AML SOPs for a detailed discussion of calibration curves.

9.3 Aqueous Sample preparation - Prepare sample using SW-846 Method 3511 (Microextraction) with EPA 552.2/552.3 modifications as described below:

9.3.1 Remove samples and standards from storage and allow them to reach room temperature.

9.3.2 Prepare calibration standards (ICAL, ICV, and CCV) as described in earlier sections.

9.3.3 Prepare batch quality control samples (MB, LCS, and LCSD) as described in earlier sections.

9.3.4 Prepare MS and MSD samples as described in earlier sections.

9.4 Soil Sample Preparation – Prepare sample using SW-846 Method 3570 (Microextraction) with modifications as described below:

9.4.1 Mark the outside of several 60-mL VOA vials with a mark at the 50-mL volume.

9.4.2 Remove the cap and weigh out 10-g of soil or sediment into the 60-mL VOA vial. Add reagent water to the mark so that the final volume of the soil and water mixture is 50-mL. Replace the cap.

9.4.3 Swirl the vial to suspend the soil/sediment in the aqueous phase. Extract the aqueous soil extract for target compounds in the same manner as aqueous samples (Section 9.5 and 9.6).

9.5 Sample Extraction

9.5.1 Place 40 mL of the water sample into a precleaned 60 mL glass vial with a Teflon-lined screw cap using a graduated cylinder. Add 20 µL of surrogate standard (10.0 µg/mL 2,3-dibromopropionic acid in MTBE. ***Note:*** When fortifying an aqueous sample with either surrogate or target analytes contained in MTBE, be sure that the needle of the syringe is well below the level of the water. After injection, cap the sample and invert once. This insures that the standard solution is mixed well with the water.

9.5.2 Adjust the pH to less than 0.5 by adding at least 2 mL of concentrated sulfuric acid. Cap, shake and then check the pH with a pH meter or narrow range pH paper.

9.5.3 Quickly add approximately 2 g of copper II sulfate pentahydrate and shake until dissolved. This colors the aqueous phase blue and therefore allows for the analyst to better distinguish between the aqueous phase and the organic phase in this microextraction.

9.5.4 Quickly add 16 g of muffled sodium sulfate and shake for three to five minutes until almost all is dissolved. Sodium sulfate is added to increase the ionic strength of the aqueous phase and thus further drive the haloacetic acids into the organic phase. The addition of this salt and the copper II sulfate should be done quickly so that the heat generated from the addition of the acid will help dissolve the salts.

9.5.5 Add 4.0 mL MTBE and place on the mechanical shaker for 30 minutes. ***Note:*** If hand-shaken, two minutes is sufficient if performed vigorously. Allow the phases to separate for approximately five minutes.

9.6 Extract Methylation

9.6.1 Using a pasteur pipet, transfer approximately 3 mL of the upper MTBE layer to a 15 mL graduated conical centrifuge tube.

9.6.2 Add 1 mL 10% sulfuric acid in methanol to each centrifuge tube. Cap the centrifuge tubes and place in the heating block (or sand bath) at 50°C and maintain for two hours. The vials must fit snugly into the heating block to ensure proper heat transfer. At this stage, methylation of the method analytes is attained.

9.6.3 Remove the centrifuge tubes from the heating block (or sand bath) and allow them to cool before removing the caps.

9.6.4 Add 4 mL saturated sodium bicarbonate solution to each centrifuge tube in 1 mL increments. Exercise caution when adding the solution because the evolution of CO_2 in this neutralization reaction is rather rapid. Shake each centrifuge tube for two minutes. As the neutralization reaction moves to completion, it is important to continue to exercise caution by venting frequently to release the evolved CO_2.

9.6.5 Transfer exactly 1.0 mL of the upper MTBE layer to an autosampler vial. A duplicate vial should be filled using the excess extract.

9.6.6 Add 10 µL of internal standard to the vial to be analyzed (25 µg/mL 1,2,3-trichloropropane in MTBE).

9.6.7 Analyze the samples as soon as possible. The sample extract may be stored up to seven days if kept at 4°C or less or up to 14 days if kept at -10°C or less. Keep the extracts away from light in amber glass vials with Teflon-lined caps.

9.7 Gas Chromatographic Analysis

9.7.1 Set up the GC as described in Section 9.1 and perform analysis of the initial calibration standards (ICAL).

9.7.2 Construct a calibration curve as described in Section 9.2. Verify the ICAL with a second-source calibration standard (ICV).

9.7.3 Prior to the analysis of any sample extracts, verify the ICAL with a continuing calibration verification (CCV) standard.

9.7.4 Inject 2 µL of the sample extract. Record the resulting peak sizes in area or height units as practiced for the ICAL.

9.7.5 If the response for the peak exceeds the working range of the system, dilute the extract, add an appropriate additional amount of internal standard and reanalyze. The analyst must not extrapolate beyond the calibration range established.

9.8 Calculations

9.8.1 Identify target compounds in the sample chromatogram by comparing the retention time of the suspect peak to retention times generated by the calibration standards and the check standard.

9.8.2 Use the calibration curve or calibration factor to directly calculate the uncorrected concentration (C_i) of each analyte in the sample (e.g. calibration factor x response).

9.8.3 Calculate the sample volume (V_s) in mL as equal to the net sample weight in grams (Section 9.8.3).

9.8.4 Calculate the corrected sample concentration in aqueous and soil samples as follows:

$$\text{Aqueous Concentration } (\mu g/L) = (C_i) * DF$$

$$\text{Soil Concentration } (\mu g/kg) = \frac{(C_i) * 50 * DF}{(W_s/1000) * (PS/100)}$$

where:
C_i is the measured extract concentration (µg/L) after dilution, if any
V_s is the aqueous sample volume (mL)
W_s is the weight of the soil sample (g)
DF is the dilution factor for the GC analysis
PS is the percent solids for the soil sample
50 is the default sample volume (mL) for soil samples

9.8.5 Report the results in µg/L (µg/kg for soil based on dry weight) to three significant figures.

10.0QUALITY CONTROL

10.1Using this SOP will involve implementing the referenced EPA method at the laboratory. Method implementation involves the following: chromatographic separation of the target analytes on two GC columns, determination of retention time windows for each compound, determination of method detection limits for each sample matrix and/or preparation method, spiked sample analysis, initial demonstration of acceptable recoveries, etc. Method implementation procedures are described on the AML SOP on MDL.

10.2Analytical data, which includes calibration data, sample preparation logs, sample data and quality control data should be gathered prior to review. A review checklist (see Appendix 5 for details) is recommended to document verification of calculations and adherence to method procedures. Exceptions should be documented. Samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on these checklists.

10.3Initial and Continuing Demonstration of Capability – Before analyzing any environmental samples, perform MDL and spike recovery studies to demonstrate laboratory capability for this analysis as described below and in applicable AML SOPs:

10.3.1 MDL Studies – See applicable AML SOPs for performing these studies.

10.3.2 Spike Recovery Studies - Prepare four replicates of the LCS by adding an appropriate aliquot of the primary dilution standard or quality control sample to reagent water. This reagent water should contain ammonium chloride at the same concentration as that specified for drinking water samples. Calculate the mean percent recovery and the standard deviation of the recoveries.

10.3.2.1 For each analyte, the mean recovery value, expressed as a percentage of the true value, must fall in the range of 80-120% and the relative standard deviation should be less than 20%.

10.3.2.2 For those compounds that meet these criteria, performance is considered acceptable and sample analysis may begin. For those compounds that fail these criteria, this procedure must be repeated using four to seven fresh samples until satisfactory performance has been demonstrated. Maintain these data on file to demonstrate initial capabilities.

10.3.3 Proficiency Tesing (PT) Samples – Analyze two sets of PT samples (e.g. WS samples from ERA or APG) initially and every 6 months thereafter to demonstrate proficiency.

10.3.4 External Quality Control Samples – These are required to be analyzed quarterly. However, the second source ICV, which is analyzed with each ICAL provides a more frequent verification of the analytical process.

10.4 Summarize sample results using Form-1 Equivalents (See Appendix 12). Summarize QC results and QC recoveries in Form-1 and Form-3 equivalents respectively for MS/MSD (Appendix 7) and LCS/LCSD (Appendix 8).

10.5 Surrogate recovery should be within QC limits (70 to 130% for aqueous samples and 50 to 150% for soil) samples, spikes and blanks. Document surrogate recoveries in Form-2 equivalents (See Appendix 9) Flag recoveries that are outside the QC limits with an asterisk (*). If re-extraction and/or reanalysis has been performed, and the surrogate recoveries for reanalysis are within QC limits, report data based on acceptable surrogate recoveries. If the surrogate recoveries are outside the QC limits for the reanalysis, report data for both analyses with surrogate recovery flags.

10.6 Method blanks (MB) should be free of target compounds. Report target compounds that are present in the MB at levels greater than the reporting limit (RL) using J qualifiers if the levels are less than MQL. The action level for method blank is one half of the MQL. See Table 1 for a list of MDLs, RLs, and MQLs. Details of MDL determinations are included in Appendix 10. Samples associated with the method blank containing target compounds at levels greater than on half the MQL will require re-extraction and reanalysis unless other criteria accepable to the client are used. Since method blanks may be contaminated when they are associated with samples containing high levels of target compounds, only samples that contain less than one tenth of the MB amount may need to be re-extracted and reanalyzed.

10.7 MDL Check Standard – This is the same as the Laboratory Performance Check Standard (LPC), which is required to be run at the beginning of an analysis set, prior to any calibration standard or sample analysis and after an initial solvent analysis. This check standard insures proper performance of the GC by evaluation of the instrument parameters of detector sensitivity, peak symmetry, and peak resolution. It furthermore serves as a check on the continuity of the instrument's performance. In regards to sensitivity, it allows the analyst to ascertain that this parameter has not changed drastically since the analysis of the MDL study.

10.7.1 Analyze a standard that is 2 times the MDL or one half of the MQL. All the analytes should be detected on this MDL Check Standard.

10.7.2 Inability to demonstrate acceptable instrument performance indicates the need for reevaluation of the instrument system. The concentrations of the LPC standard may be adjusted to be compatible with the laboratory MDLs and MQLs.

10.7.3 If column or chromatographic performance cannot be met, one or more of the following remedial actions should be taken. Break off approximately 1 m of the injector end of the column and re-install, install a new column, adjust column flows or modify the oven temperature program.

10.7.4 Perform a new MDL study, if a successful MDL check standard cannot be analyzed.

10.8 LCS recoveries should be within QC limits (70-130 for water) for the target compounds. Flag recoveries that are outside the QC limits with an asterisk (*). See

Table 1 for QC limits. Data qualification, re-extraction, and/or reanalysis of associated samples may be necessary depending on project requirements, turnaround time and other constraints.

10.9 MS recoveries should be within QC limits (70-130 for water) for the target compounds. Flag recoveries that are outside the QC limits with an asterisk (*). See Table 1 for QC limits. Data qualification, re-extraction, and/or reanalysis of associated samples may be necessary depending on project requirements, turnaround time and other constraints.

10.10 Initial Calibration Curves

10.10.1 Procedural Standard Calibration, which is employed in this SOP requires that aqueous calibration standards be prepared and processed (e.g., purged, extracted and/or derivatized) in exactly the same manner as a sample. All steps in the process are included in the calibration. Using procedural standard calibration compensates for any inefficiencies in the processing procedure. As a result, variation for the calibration curve is expected to be greater than those that employ calibration standards that have not gone through the sample preparation process.

10.10.2 Linear or non-linear regression methods may be used to generate calibration curves.

10.10.3 Alternately, an average relative response factor (RRF) can be calculated and used for quantitation. Relative response factors are calculated for each analyte at each of the five concentration levels. If the mean RRF value over the working range is constant (<20% RSD), the RRF can be assumed to be invariant and the average RRF used for calculations.

10.11 The CCVs for this method are identical to the LCS/LCSD samples. The CCV recoveries should be within QC limits (70-130 for water) for the target compounds.

10.11.1 At least one CCV must be extracted with each set of samples. The LCS extracts may be used as CCVs. A CCV must be analyzed at the beginning of each analysis set, after every tenth sample analysis and after the final sample analysis, to ensure that the instrument is still within calibration.

10.11.2 Calculate analyte recoveries for all target analytes. In order for the calibration check to be considered valid and subsequently for the preceding ten samples to be considered acceptable with respect to calibration, recoveries must fall between 70% and 130% for all the target analytes.

10.11.3 CCVs need not necessarily be different extracts but can be injections from the same extract, including the LCS extracts as long as the sample holding time requirements are met.

10.11.4 If this criteria cannot be met, the continuing calibration check standard extract is re-injected in order to determine if the response deviations observed from the initial analysis are repeated. If this criteria still cannot be met, a second CCV should be extracted and analyzed or a CCV that has already been analyzed and has been found to be acceptable should be run. If this second CCV fails, then the instrument is considered out of calibration and needs to be recalibrated.

10.12 Internal Standards

10.12.1 The analyst must to monitor the IS response (peak area or peak height) of all injections during each analysis day. A mean IS response should be determined from the five-point calibration curve. The IS response for any run should not deviate from this mean IS response by more than 30%. It is also acceptable if the IS response of an injection is within 15% of the daily continuing calibration standard IS response.

10.12.2 If a deviation greater than this occurs with an individual extract, optimize instrument performance and inject a second aliquot of that extract.

10.12.2.1 If the reinjected aliquot produces an acceptable internal standard response, report results for that aliquot.

10.12.2.2 If a deviation of greater than 30% is obtained for the reinjected extract, the analyst should check the calibration by analyzing the most recently acceptable CCV. If the CCV fails the ±30% criteria, recalibration is in order. If the CCV is acceptable, analysis of the sample should be repeated, provided the sample is still available. Otherwise, report results obtained from the reinjected extract as an estimated value (J qualification).

10.13 Report sample results on Form-1 equivalents (See Appendix 12). Report target compounds that are present at levels greater than the reporting limit (RL) using "J" qualifiers if the levels are less than MQL. Compounds that were also detected in the associated MB should also be qualified with a "B" code. If the concentration of any of the target compounds exceed the upper limit of calibration (ICAL), flag the affected compounds data with an estimated or "E" flag. The extracts may need to be diluted and reanalyzed until the concentrations are within the calibration limits for normal turnaround analyses. For rapid turnaround analyses sample results may be reported with "E" qualifiers if sufficient time is not available for reanalysis.

11.0 **DATA VERIFICATION AND VALIDATION -** This section will provide guidance and procedures to assess if data produced using this SOP is valid and legally defensible. Document review and comments on or more of the the checklists (See Appendix 5 for details). Verify that sample preparation logs are complete and the instrument run logs are available for review (See Appendix 11 for details). Anomalies and exceptions should be documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on the checklists. Army (USACE) and Air Force (AFCEE) projects may have special requirements (See Appendix 13) that will require extra attention. Requirements may also be from the proposed implementation of the Department of Defense Quality Systems Manual (QSM) for Environmental Laboratories (Final Revision 2, June 2002), that will govern analyses performed for Army, Navy, Air Force, and the Marine Corps. A simple, universal procedure for evaluating and choosing appropriate corrective action doe not exist to deal with situations when one or more QC parameters are outside the acceptance window. The

objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints.

11.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) projects may have additional requirements that are related to USACE laboratory validation (CX), USACE District requirements, government contract requirements, specific USACE project requirements or USACE Architect/Engineer (A/E) requirements. USACE-CX requirements (Summary of Method Quality Objectives for Method 8011) are included in Appendix 13.

11.1.1 The USACE and/or AFCEE requirements shown in Appendix 13 and elsewhere in this SOP are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may or may not be attainable for routine sample analysis. Since USACE MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

11.1.2 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality objectives and/or other QC requirements. Exceptions and data qualifications necessary should be documented on the checklists and eventually on case narratives.

11.1.3 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist, perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete. For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete. For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director will review the checklists (initial/date as necessary), prepare case narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

11.2HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

11.2.1 WATER - The holding times for sample extraction is 14 days from sampling. The holding time for instrumental analysis is for 7 days from extraction, if the extracts are refrigerated (4˚C) and 14 days if they are kept froze (-10˚C).

11.2.2 SOIL – The soil holding times are unavailable. It is recommended that the water holding times be used for the soil matrix.

11.2.3 Holding times are established by comparing the chain of custody sampling date with the date of the injection.

11.2.4 ACTION – If holding times have been exceeded, contact the client. All positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or innvalid (I).

11.3INITIAL CALIBRATION – Instrument calibration (See Appendix 3 for details) criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the GC/ECD is capable of producing data within a range that is linear. The ICAL includes initial verification (ICV) of the ICAL with a second source standard for USACE and AFCEE projects (See Appendix 13 for details).

11.3.1 Compounds – Check to see if any other compounds are outside the criteria of 20% or less for %RSD. If a compound is outside the 20% criteria, check to see if linear regression was used. The limit for "r" used in linear regression is 0.990.

11.3.2 ACTION – Positive results for compounds not meeting the criteria may need to be qualified as estimated (J) by the user (client). Report detects and non-detects without qualifiers and mention these outliners in the case narrative.

11.4CONTINUING CALIBRATION – The calibration verification consists of two parts: (a) Initial calibration verification (ICV), which is performed using a second source standard to check on the ICAL standard; and (b) Continuing calibration verification (CCV), which is used to check that the instrument is operating under the linear range of the initial calibration. The CCV is also used to check the stability of the machine and to verify that the instrument is still operating under the linear range of the initial calibration and has remained stable every 12 hours.

11.4.1 Compounds – Check to see if the %D for any compounds are outside the criteria of 30% or less.

11.4.2 ACTION – Positive results for compounds not meeting the criteria may be qualified as estimated (J) by the user (client). Report non-detects without qualifiers and mention these outliners in the case narrative.

11.5METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Sample data qualification to alert blank contamination is performed by the laboratory. Sample data qualification based on all the blank results (laboratory MB, equipment blank, and other field blanks) is performed by the user (client). The processing of blanks helps eliminate reporting of false positives in project reports.

11.5.1 Method blanks should be free of all target anaytes and other interfering non-target analytes. To be acceptable, blank results should not exceed one half of the applicable MQL for any compound.

11.5.2 Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples. If levels greater than one half the MQLs are present, inspect and correct the problem. Reanalyze all associated samples. Contact client if other criteria are applicable. The only exception may be quick turn around projects.

11.5.3 ACTION – All positive results are reported with a “B” code, if contaminants are found in the method blank.

11.5.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, then no action is taken.

11.5.5 ACTION – Any compound detected in the sample which were also detected in associated method blank at levels greater than one half the MQLs should be re-extracted unless other client criteria are applicable.

11.5.6 ACTION – If contaminants resulting in GC/ECD peak saturation are present, all affected compounds are qualified as invalid (I).

11.6SURROGATE RECOVERIES – Surrogates or system monitoring compounds are different structurally, but similar chemically to classes of target compounds. These surrogate compounds are added just prior to extraction of the samples and the percent recoveries of the surrogates are calculated and evaluated.

11.6.1 Verify that the recovery (See Appendix 9 for details) of the surrogate compound is within QC limits (70% to 130% in water and 50 to 150% in soil). Flag outliers with an asterisk (*).

11.6.2 ACTION – When surrogate recoveries are outside control limits the sample must be re-analyzed. Reanalysis may be performed under dilution if original analysis should indicate high levels of target and non-target analytes. Reextraction and cleanup will be necessary if reanalysis does not resolve the problem. If surrogate recoveries are still outside the limits, report both sets of data and described the problem in the case narrative.

11.7LABORATORY CONTROL STANDARD (LCS) – A LCS analysis is used to evaluate method competence in the absence of matrix effects. The LCS should be from a second source standard. The LCS may provide a mechanism to check method efficiency for target compounds without the sample matrix as a factor.

11.7.1 LCS and LCSD recoveries (See Appendix 8 for details) should be within QC limits (70 to 130).
11.7.2 Check the results for the LCS recoveries to see if the results are within the acceptable range. Flag outliers with an asterisk (*).
11.7.3 ACTION – If deviations are noticed, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements. Check with the project manager about such requirements. No action may need to be taken on LCS and/or LCSD recovery data alone either to reanalyze samples and/or to qualify associated samples for most projects. Professional judgment may be used by the user in evaluating sample data qualification options in conjunction with other QC data for the project.

11.8MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike data (See Appendix 7 for details) are used to evaluate the precision and accuracy of the analytical method on project samples.

11.8.1 The MS/MSD percent recoveries sould be within QC limits (70 to 130).
11.8.2 Check the results for the MS/MSD recoveries. Flag outliers with an asterisk (*).
11.8.3 Perform one manual calculation using raw data and extraction sheet information.
11.8.4 ACTION – No action is taken on MS/MSD recoveries data alone to qualify all associated samples. A general rule is to have at least 90% of the target analytes within the control limits. Since matrix interference could lead to lead to serious analytical problems, the supervisor should be consulted when significant outliers are noted. Document QC outliers for %recovery and RPD on the checklist and on client reports (*) and draw attention to the QC outliers on the case narrative. No action may be taken on MS/MSD recovery data alone either to reanalyze samples and/or to qualify associated samples either by the analyst or by the end user. Professional judgment may be used by the user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

11.9INTERNAL STANDARDS

11.9.1 The analyst must to monitor the IS response (peak area or peak height) of all injections during each analysis day. A mean IS response should be determined from the five-point calibration curve.
11.9.2 The IS response for any run should not deviate from this mean IS response by more than 30%.

11.9.3 It is also acceptable if the IS response of an injection is within 15% of the daily continuing calibration standard IS response.

11.9.4 ACTION - If a deviation greater than this occurs with an individual extract, optimize instrument performance and inject a second aliquot of that extract. If the reinjected aliquot produces an acceptable internal standard response, report results for that aliquot.

11.9.5 ACTION - If a deviation of greater than 30% is obtained for the reinjected extract, the analyst should check the calibration by analyzing the most recently acceptable CCV. If the CCV fails the ±30% criteria, recalibration is in order. If the CCV is acceptable, analysis of the sample should be repeated, provided the sample is still available. Otherwise, report results obtained from the reinjected extract as an estimated value (J qualification).

11.10 TARGET COMPOUND IDENTIFICATION – Criteria for GC/ECD qualitative analysis is to minimize the number of erroneous positive or negative identifications of compounds.

11.10.1 Positive compounds must be within +/- 0.06 relative time (RRT) units of the standard RRT. The results should be >MDL or other threshold for reporting positives (e.g. LLR, RL) on both columns.

11.10.2 The RPD for sample amounts from the two columns for identified compounds should be within <40%. Document RPD calculations (See Appendix 6 for details) in Sample Result worksheets (Form-10 equivalents).

11.10.3 ACTION - If the RPD for positive results are <40%, report the result from the primary column without any flag.

11.10.4 ACTION - If the RPD is >40%, report the result from the primary column with a “J” flag. The result from the confirmation column may be reported with a “J” flag at the discretion of the analyst, if intererence is suspected for the compound of interest in the primary column.

11.10.5 ACTION – Professional judgement must be used when applying the criteria for GC/ECD analysis of target analytes. Take into account factors such as matrix effect and column bleed.

12.0 REFERENCES

12.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8011, 1,2-Dibromoethane and 1,2-Dibromo-3-chloropropane by Microextraction and Gas Chromatogrpahy, July 1992.

12.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.

12.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

12.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3500B, Organic Extraction and Sample Preparation, December 1996.

12.5 J.W. Hodgeson, J. Collins, and R.E. Barth, EPA Method 552.0, Determination of Haloacetic Acids and Dalapon in Drinking Water by Liquid-Liquid Extraction, Derivatization, and Gas Chromatogrpahy with Electron Capture Detector, (1990).

12.6 J.W. Hodgeson, and D. Becker, EPA Method 552.1, Determination of Haloacetic Acids and Dalapon in Drinking Water by Liquid-Liquid Extraction, Derivatization, and Gas Chromatogrpahy with Electron Capture Detector, (1992).

12.7 D.J. Munch and J.W. Munch, EPA Method 552.2, Determination of Haloacetic Acids and Dalapon in Drinking Water by Liquid-Liquid Extraction, Derivatization, and Gas Chromatogrpahy with Electron Capture Detector, Revision 1.0(1995).

12.8 M.M. Domino, B.V. Pepich, D.J. Munch, and P.S. Fair, EPA Method 552.3, Determination of Haloacetic Acids and Dalapon in Drinking Water by Liquid-Liquid Extraction, Derivatization, and Gas Chromatogrpahy with Electron Capture Detector, EPA 815-B-03-002, Revision 1 (2003).

12.9 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3511, Organic compounds in water by microextraction, Revision 0, November 2002.

12.10 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3570, Microscale Solvent Extraction (MSE), Revision 0, November 2002.

12.11 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.

12.12 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

12.13 AML Standard Operating Procedures, Q-Water-RC.

12.14 AML Standard Operating Procedures, Q-WasteMgmt-RD.

12.15 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

12.16 AML Standard Operating Procedures, Q-Controlcharts-RC.

Table 1
Aqueous Sample MDL, LLR, MQL and QC Limits for LCS and MS

Matrix: Water	Preparation Method	EPA 552.2
	Analytical Method	EPA 552.2

Compound	Spike Amount ug/L	MDL Column No. 1 ug/L	MDL Column No. 2 ug/L	**MDL Method ug/L A**	**MQL ug/L C**	**Ratio MQL to MDL C/A**	**LCS/MS 3-sigma Control Limits** Lower **%R**	Upper **%R**	**LCS/MS 4-sigma ME Limits** Lower **%R**	Upper **%R**	**RPD** Limit %
Monochloroacetic acid	1.50	0.419	0.901	**0.901**	3.0	3.3	70	130	60	140	50
Monobromoacetic acid	1.00	0.475	0.391	**0.475**	2.0	4.2	70	130	60	140	50
Dichloroacietic acid	1.50	0.545	0.827	**0.827**	3.0	3.6	70	130	60	140	50
Dalapon	1.00	0.433	0.514	**0.514**	2.0	3.9	70	130	60	140	50
Trichloroacetic acid	0.50	0.310	0.283	**0.310**	1.0	3.2	70	130	60	140	50
Bromochloroacetic acid	1.00	0.568	0.537	**0.568**	2.0	3.5	70	130	60	140	50
Dibromoacetic acid	0.50	0.208	0.265	**0.265**	1.0	3.8	70	130	60	140	50
Bromodichloroacetic acid	1.00	0.388	0.548	**0.548**	2.0	3.7	70	130	60	140	50
Chlorodibromoacetic acid	2.50	0.966	0.759	**0.966**	5.0	5.2	70	130	60	140	50
Tribromoacetic acid	5.00	3.122	2.972	**3.12**	10.0	3.2	70	130	60	140	50
Surrogate	6.00				Mean:	3.8	70	130	60	140	

The 3-sigma control limits are from EPA Method 552.2.
The marginal exceedence (ME) limits (4-sigma) are based on recommendations contained in the DoD Quality Systems Manual.

Table 2
List of Appendices

Appendix Number	Appendix Topic
1	Chromatography Conditions
2	Retention Time Summary
3	Initial Calibration (ICAL) Summary (Form-6 equivalents) Instrument Printouts
4	Calibration Verification (ICV and CCV) Summary (Form-7 equivalents) Instrument Printouts and Standard Chromatograms
5	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
6	Sample Result Worksheets (Form-10 equivalents)
7	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
8	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
9	Surrogate Recovery Reports (Form-2 equivalents)
10	Method Detection Limit (MDL) Study Summary
11	QC Association Forms (Form-4 equivalents) Sample Preparation Logsheets Instrument Run Log
12	Sample Reports (Form-1 equivalents)
13	Method Quality Objectives for Method 8000B and/or 8011 (USACE, AFCEE, DoD QSM, etc.)

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. O-Herbicides-RC

CHLOROPHENOXY HERBICIDES BY GC/ECD (EPA 515.1, 515.4, and SW846-8151A)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph. D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP), which is based on EPA methods (515.1, 515.4 and SW-846 Method 8151A) is used to determine the concentrations of various chlorophenoxy herbicides in solid and liquid matrices, using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD). This SOP is normally used for the determining the following chlorinated acid herbicides and related compounds in groundwater, wastewater, soil, sediment and waste samples. The words, method and SOP are used synonymously in this document.

Compound	**CAS Number**
2, 4-D	94-75-7
2, 4-DB	94-82-6
2, 4, 5-TP (Silvex)	93-72-1
2, 4, 5-T	93-76-5
3,5-Dichlorobenzoic acid	51-36-5
Acifluorfen	50594-66-6
Bentazon	25057-89-0
Chloramben	133-90-4
Dacthal di-acid	
Dalapon	75-99-0
Dicamba	1918-00-9
Dichlorprop	120-36-5
Dinoseb	88-85-7
MCPA	94-74-6
MCPP	93-65-2
Pentachlorophenol	87-86-5
Picloram	1918-02-1

Because these compounds are produced and used in various forms (acid, amine, salt, ester, etc.) this SOP includes a hydrolysis step to convert the various forms to the acids form prior to derivatization such as methylation. The results therefore correspond to the total amount of herbicide compounds in the samples. Other compounds that may be analyzed by this method are 4-nitrophenol, pentachlorophenol, acifluorfen, bentazon, chloramben, DCPA diacid, 3, 5-dichlorobenzoic acid, 5-hydroxydicamba and picloram. These compounds are not analyzed on a daily basis. Therefore, method performance should be established before data can be reported for unknown samples.

1.2 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatography and in the interpretation of gas chromatograms. Only experienced analysts should be allowed to work with diazomethane due to health hazards (explosive, carcinogen) associated with this compound.

2.0 SUMMARY OF METHOD

2.1 Water (One liter) samples are acidified and extracted with ether. Soil samples (Thirty grams) are acidified, extracted with successive acetone and ether steps. Waste (One gram) is diluted with ether. The ether extracts from the various matrices is then hydrolyzed with potassium hydroxide, solvent washed with ether, acidified and re-extracted with ether. The ether extract is concentrated, derivatized with diazomethane, and solvent exchanged to hexane. Microextraction procedures can be used when detection limit requirements are met.

2.2 Analysis is by gas chromatography with dual capillary columns and electron capture detectors with internal standard calibration. The herbicides are reported as acid equivalents. The sensitivity of the method is usually determined by the level of interferences.

3.0 INTERFERENCES

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in the gas chromatograms. Method blanks are extracted with each extraction set to monitor interferences.

3.2 Glassware should be cleaned following normal cleaning procedures. Since herbicides are strong organic acids and react readily with alkaline active sites, all glassware used in the extraction procedure need further cleaning. Glassware should be rinsed with 20% HCl followed by an acetone rinse immediately before use. Glasswool should be acid rinsed as well. Anhydrous sodium sulfate used for drying soil samples should be acidified before use.

3.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from sample to sample. Organic acids, especially chlorinated acids, cause the most direct interference with this method. Phenol, including chlorophenols, chlorinated hydrocarbons, phthalate esters and oils also cause interferences. The alkaline hydrolysis and subsequent ether wash of the basic solution removes many of these interferences.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph - A Hewlett Packard 5890 Series II GC equipped with dual Ni-63 electron capture detectors is used. The system is complete with a temperature-programmable GC for splitless injection. The system includes all accessories, including syringes, analytical columns and gases. The column flow rate remains constant throughout the temperature program operation.

4.2 Gas chromatographic columns - A dual column system is employed. A 30-meter x 0.32-mm ID capillary column (primary) coated with DB-5 for a film thickness of 0.25-um (J&W Scientific) is employed. A 30-meter x 0.32-mm ID capillary column (confirmation) coated with DB-1701 for a film thickness of 0.25-um (J&W Scientific) is employed. Equivalent columns may be used in place of both columns. The primary and confirmation columns are used interchangeably.

4.3 Data system - Hewlett Packard Environquant is used for data acquisition and storage on machine-readable media of all data obtained throughout the duration of the GC program.

4.4 Microsyringes - 10, 25, 100, 250, 500 and 1000-uLs.

4.5 Balance - Analytical, 0.0001 grams and top loading, 0.1 grams.

4.6 Disposable pipettes - Pasteur

4.7 Test tubes - 5-ml to 8-ml, with Teflon lined screw caps.

4.8 Separatory funnels - 2000- mL

4.9 Kuderna-Danish (K-D) apparatus as described below:

- 4.9.1 KD Tubes
- 4.9.2 KD Evaporation flask (500-mls), attached to concentrator tubes
- 4.9.3 Three ball macro Snyder column
- 4.9.4 Clamps

4.10 Boiling Chips - solvent extracted

4.11 Heated water bath (60º to 65ºC)

4.12 Microsyringes - 10, 50, 100, 500, 1000-uLs.

4.13 Tekmar Sonic Disrupter

4.14 Glass wool - acid rinsed and dried

4.15 Stainless steel table spoons

4.16 Glassware and kit for the preparation of the diazomethane solution

4.17 Various beakers, funnels, 125-ml Erlenmeyer flasks, 24/40 ground glass 250-ml and 500-ml Erlenmeyer flasks

4.18 pH paper – 0-14 range

4.19 Mechanical shaker

4.20 Centrifuge

5.0 REAGENTS

5.1 It is assumed that all inorganic/organic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where available. Other reagents should be of high enough purity to maximize the accuracy of the determination.

5.2 Organic free reagent water - All references to water in this method should be organic free reagent water.

5.3 Solvents – Solvents used should be pesticide grade or equivalent

- 5.3.1 Diethyl ether, unpreserved and free of peroxides.
- 5.3.2 Methylene chloride
- 5.3.3 Acetone
- 5.3.4 Isooctane
- 5.3.5 Toluene

5.3.6 Carbitol –(diethylene glycol monoethyl ether)
5.3.7 Hexane

5.4 Sodium Sulfate – granular, anhydrous. Acidify with concentrated sulfuric acid and preclean with methylene chloride. Mix 1-kg of sodium sulfate (precleaned with methylene chloride) with 300-mls acetone and 1-ml conc. sulfuric acid. Dry at 50 degrees C. Check by adding 1-g of acidified sodium sulfate to 5-mls of water. The pH should be < 4.

5.5 Acid solutions

5.5.1 1:1 sulfuric acid – slowly add 50-mls conc. sulfuric acid to 50-mls of water.
5.5.2 1:3 sulfuric acid – slowly add 250-mls conc. sulfuric acid to 750-mls of water.
5.5.3 20 % HCl – slowly add 200-mls conc. HCl acid to 800-mls of water.
5.5.4 5% acidified sodium sulfate – dissolve 50-grams acidified sodium sulfate in 1-liter of water.

5.6 Potassium Hydroxide – 37% aqueous solution. Dissolve 185-grams potassium hydroxide pellets in 500-mls of water.
5.7 Diazald (n-methyl-n-nitroso-p-toluenesulfonamide)
5.8 Diazald Kit (or equivalent) – Used to prepare diazomethane solution (See Appendix 1 for diazomethane preparation).
5.9 Silicic acid – 100-mesh
5.10 Stock Standard solutions – The stock standards for instrument calibrations are purchased as certified solutions from Ultra Scientific (Catalog number HBM-8150M). All the compounds in the stock standard are in the methylated form. The concentration of each analyte (or compound) is listed in Table I. After opening the stock vial, transfer the stock solutions to a GC vial and seal with PTFE-lined screw caps. Store at 4 degrees C and away from light. Stock standards expire on the date shown by the manufacturer. Check stock standards for signs of evaporation or degradation, especially immediately prior to preparing calibration standards from them.
5.11 Intermediate Standard Solutions - From the Stock solution, prepare an intermediate solution by diluting the stock solution by a factor of 1:100. Take 1.0-ml of the stock solution and dilute it to 100-mls with hexane in a volumetric flask. The concentration of each compound in the intermediate solution, which is also used as the highest concentration calibration standard, is shown in Table II. The intermediate standard solution should be replaced after one year or sooner if routine QC or comparisons with check standards indicate problem(s).

5.12 Calibration standard solutions – A five-point initial calibration (ICAL) curve is prepared from the intermediate standard solution. Calibration solutions should be replaced after six months or sooner if comparisons with check standards indicate a problem.

5.12.1 The intermediate solution also serves as the highest point of the curve (herbicide Std 5). Prepare four separate dilutions of the intermediate solution using the following tables to prepare a set of five calibration solutions:

5.12.2 MID HIGH POINT: Take 700-uls of the intermediate solution and dilute with 300-uls of hexane. This is herbicide Std 4 (Mid high point). The concentrations of the analytes in this solution are shown in Table III.

5.12.3 MID POINT: Take 500-uls of the intermediate solution and dilute with 500-uls of hexane. This is herbicide Std 3 (Mid point), which is described in Table IV. This solution will also serve as standard used to verify calibration (CALV) or as the continuing calibration verification standard (CCV).

5.12.4 LOW MID POINT: Take 300-uls of the intermediate solution and dilute with 700-uls of hexane. This is herbicide Std 2 (Low-Mid point), which is described in Table V.

5.12.5 LOW POINT: Take 100-uls of the intermediate solution and dilute with 900-uls of hexane. This is herbicide Std 1 (Low point), which is described in Table VI.

5.13 Surrogate solution – The compound 2, 4-Dichlorophenylacetic acid (DCAA) is used as the surrogate or system monitoring compound in this method. A shooter solution is purchased from Ultra Scientific (Catalog number PPS-164X) at a concentration of 2-ug/ml in methanol. Spike 0.5-ml of this solution into every sample including all the QC samples. Stock surrogate standards expire on the date shown by the manufacturer. Check stock standards for signs of evaporation or degradation, especially immediately prior to preparing calibration standards from them.

5.14 Matrix Spiking Solution – The MS/MSD spiking stock solution is purchased from Ultra Scientific (Catalog number HBM-8150A). The Initial concentration is the same as in the calibration stock solution (Table I) except that the compounds are in the acid form. This stock solution is diluted by a factor of 1:40 with acetone to prepare MS/MSD spiking solution. Take 1.25-mls of the stock solution and dilute to 50-mls with acetone in a volumetric flask. Spike 1.0-ml of this solution to the MS/MSD samples. When the volume of the final extract is 5-mls, the final concentrations of the analytes are shown in Table VII. Stock MS/MSD standards expire on the date shown by the manufacturer. The working MS/MSD spiking solutions should be replaced after six months or sooner if comparisons with check standards indicate a problem.

5.15 Laboratory Control Standard Spiking Solution – The LCS solution is purchased from Supelco (Catalog number 46861-U). The initial concentration of the solution is the same as the calibration stock standards except that the compounds are in the acid form. This stock solution is diluted by a factor of 1:40. Take 1.25-mls of the stock solution and dilute to 50-mls with acetone in a volumetric flask. Spike 1.0-ml of this solution to the LCS. When the final extract is at a final volume of 5-mls, the final extract concentrations will be as shown in Table VIII. Stock LCS/LCSD standards expire on the date shown by

the manufacturer. The working LCS/LCSD spiking solutions should be replaced after six months or sooner if comparisons with check standards indicate a problem.

5.16 Internal Standard solution – The internal standard is 4, 4' – dibromooctafluorobiphenyl (DBOB) and is purchased from Ultra scientific (Catalog number PPS-173) at a concentration 1000-ug/ml. Spike 10-uls of this solution into each 1-ml aliquot. This gives a final concentration of 10-ug/ml in the final extracts used in instrumental analysis. Fortify calibration standards and sample extracts including dilutions with the internal standard immediately prior to analysis. Stock internal standard solutions expire on the date shown by the manufacturer.

6.0 SAMPLE PRESERVATION HANDLING & STORAGE

6.1 One liter amber glass bottles with Teflon lined screw caps may be used for collecting aqueous samples in accordance with EPA SW-846 method 8081A. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 2 bottles for each water sample to be collected. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.

6.2 One 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples in accordance with EPA SW-846 method 8081A. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each solid sample. Quality control samples such as MS and MSD should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. These samples may be shared with samples collected for other analyses such as semivolatiles (8270C), PCBs (8082), Diesel (DRO, 8015-mod.) and other pesticides (8140, etc.).

6.3 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Analytical data generated outside of the specified holding times must be considered to be minimum values only. Such data may be used to demonstrate that a waste is hazardous where it shows the concentration of a constituent to be above the regulatory threshold but cannot be used to demonstrate that a waste is not hazardous. The holding time for the extraction of aqueous samples, which includes TCLP leachates is 7 days from the date of sample collection or TCLP leachate preparation. The holding times for the extraction of soil, sediment and waste samples, which includes TCLP leachate preparation is 14 days from the date of sample collection. The holding time for the instrumental analysis of sample extracts is 40 days from the date of extraction. Sample extracts should be stored at 4° C and protected from light.

7.0 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control contain a number of regulated compounds at high concentrations. The toxicity of these compounds present in such complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

7.5 Diazomethane is a carcinogen and can explode under certain conditions. When preparing the diazomethane solution use a hood, do not heat above 90 degrees C. Do not use ground-glass joints, sleeve bearings or glass stir rods. Store away from alkali metals. Solutions of diazomethane decompose rapidly in the presence of solid materials such as copper powder, calcium chloride and boiling chips.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Aqueous and soil samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters after the solvent(s) have been evaporated off in a hood.

9.0 PROCEDURE

9.1 Extraction of Water Samples

9.1.1 Mark the meniscus of the one liter bottle with a black magic marker. Pour the entire contents of the bottle into of separatory funnel. Add 150-mls of ether to the sample bottle and shake the bottle lightly. Pour the ether from the bottle into the separatory funnel of the sample. Fill the bottle with water to the meniscus mark. Pour this water into a graduated cylinder and record the amount in the extraction log book. Spike the samples with the appropriate surrogate solution. Remember to spike the MS/MSD and LCS with the surrogate as well as the appropriate spiking solution for the MS/MSD and LCS. Adjust the pH of the samples to less than 2 with 1:1 sulfuric acid.

9.1.2 Extract the samples by shaking the funnel for 2-minutes with periodic venting to release excess vapor pressure. Allow the organic layer to separate from the water for 10-minutes. If an emulsion forms more than one third the solvent volume, attempt to break it up with a stirring rod. Leave the emulsion in the water portion until the third extraction. After the third extraction, if more than one third is still emulsion, that extract may be frozen to separate the phases. Drain the water phase into a 2-liter Erlenmeyer flask. Collect the ether extract in a 250-ml Erlenmeyer flask.

Approximately 80-mls of the ether will remain dissolved in the water phase.

9.1.3 Pour the approx. 1-liter water sample back into the separatory funnel. Extract the sample two more times using 50-mls of ether each time. Use the 50-ml of ether to rinse the Erlenmeyer flask before adding it to the 2-liter separatory funnel. Combine the ether extracts in the 250-ml Erlenmeyer flask.

9.1.4 Hydrolysis – To the 250-ml Erlenmeyer flask containing the ether extracts, add 2-mls of the 37% KOH, 15-ml reagent water and Teflon boiling chips. Attach a three-ball Snyder column. Prewet the Snyder column by adding 1-ml of ether to the top. Place in a hot water bath (60-65 degrees C). The ether will evaporate in about 15-minutes leaving the basic water. Continue to heat for a total of 60-minutes. Remove the extracts and let them stand at room temperature for 10-minutes before removing the Snyder columns.

9.1.5 Solvent Cleanup – Quantitatively transfer the basic water solution to a 125-ml separatory funnel. Check the pH. It should be >12. Add more 37% KOH if the pH is below 12. Wash the basic solution twice by shaking it for 1-minute with 20-ml portions of ether. Drain the water solution back into the 250-ml Erlenmeyer. Discard the ether phase. Pour back the water solution using the 20-ml of ether to rinse the 125-ml funnel. The herbicides will remain in the basic water solution as potassium salts.

9.1.6 Re-extraction with ether – Quantitatively transfer the basic water solution back to the 125-ml sep. funnel. Acidify the solution to a pH 2 by adding 2-ml of cold 1:3 sulfuric acid. Check the pH. Add 20-ml of ether and extract the solution by vigorously shaking the funnel for 2-minutes. Drain the water layer into a 125-ml flask. Drain the ether layer into a 250-ml 24/40 ground glass neck flask. Extract the water solution two more times with 10-ml portions of ether. Use the 10-ml portion of ether to rinse the 125-ml funnel combining the extracts in the 250-ml 24/40 flask. Add sufficient acidified sodium sulfate to each extract so that some remains free-flowing. Stopper and allow the extracts to remain in contact with the sodium sulfate at least 4-hours or refrigerate overnight.

9.1.7 Concentrate – Concentrate the extract by a Kuderna-Danish apparatus and N-Evap. Transfer the ether extract into the receiver through a funnel plugged with acid washed glass wool and topped with acidified sodium sulfate. Use liberal washings with ether to complete the transfer. Use a glass rod to crush any caked sodium sulfate and rinse with ether into the receiver. Concentrate the extract to approx. 3-ml.

9.1.8 Methylation – The methyl esters can be formed with diazomethane by adding a diazomethane solution. Add 0.1-ml of MeOH to each extract and dilute with 1-ml of ether. Slowly add 2-mls of diazomethane solution to each extract. Let the extract stand for 10-minutes with occasional swirling. The yellow color must persist during this 10-minute process.

9.1.9 Solvent exchange – N-Evap the extract to 1-ml using a gentle stream of nitrogen. Add 1-ml of hexane and continue to nitrogen blow down. Do not let the extract go dry. Continue this process three more times. The yellow color will disappear during the blow-down. Add 0.1-grams of silicic acid to the extract. Dilute the extract to a final volume of 5-ml with hexane.

9.2 Extraction of soil, sediment or other solid samples.

9.2.1 Weigh approx. 30-grams of wet sample into a 500-ml wide mouth Erlenmeyer flask with a 32 ground glass neck. Add all spiking solutions accordingly. Acidify the samples with conc. HCL (2-3 mls) to a pH of 2. Allow the samples to stand for 15-minutes occasionally stirring until the pH remains below 2. Add more acid if necessary.

9.2.2 Add 20-ml of acetone to each sample, stopper and place on a shaker for 20-minutes. Add 80-mls of ether to each sample and shake for another 20-minutes.

9.2.3 Remove the flasks from the shaker and allow the solids to settle. Decant the solvent layer into a 100-ml flask. If the extraction does not have a decanted volume of at least 75-ml, an additional extraction is necessary. Pour the extracts into a 2-liter separatory funnel.

9.2.4 Check the pH of the sample to ensure that the pH remains at 2. If the pH is not at 2, add more HCl until it is stabilized. Perform two more extractions of the sample as in 9.2.2 and collect the extracts in the flasks.

9.2.5 Add the extracts and 250-mls of 5% acidified sodium sulfate solution to 2-liter separatory funnel. Gently mix the contents of the funnels for 1-minute and allow the phases to separate. Drain the water/acetone phase into 500-ml flasks and the ether phase into a separate 500-ml flask with 24/40 ground glass necks. Pour back the water/acetone phase and re-extract with 25-mls of ether. Combine the ether extracts.

9.2.6 Hydrolysis - To the 500-ml Erlenmeyer flask containing the ether extracts, add 5-mls of the 37% KOH, 30 ml reagent water and Teflon boiling chips. Attach a three-ball Snyder column. Prewet the Snyder column by adding 1-ml of ether to the top. Place in a hot water bath (60-65 degrees C). The ether will evaporate in about 20-30 minutes leaving the basic water. Continue to heat for a total of 90-minutes. Remove the extracts and let them stand at room temperature for 10-minutes before removing the Snyder columns.

9.2.7 Solvent Cleanup - Quantitatively transfer the basic water solution to a 125-ml separatory funnel. Check the pH. It should be >12. Add more 37% KOH if the pH is below 12. Wash the basic solution twice by shaking it for 1-minute with 40-mls portions of ether. Drain the water solution back into the 500-ml Erlenmeyer. Discard the ether phase. Pour back the water solution using the 20-ml of ether to rinse the 125-ml funnel. The herbicides will remain in the basic water solution as potassium salts.

9.2.8 Re-extract with ether - Quantitatively transfer the basic water solution back to the 125-ml Sep funnel. Acidify the solution to a pH 2 by adding 5

ml of cold 1:3 sulfuric acid. Check the pH. Add 40-ml of ether and extract the solution by vigorously shaking the funnel for 2-minutes. Drain the water layer into a 125-ml flask. Drain the ether layer into a 250-ml 24/40 ground glass neck flask. Extract the water solution two more times with 20-ml portions of ether. Use the 20-ml portion of ether to rinse the 125 ml funnel combining the extracts in the 250-ml 24/40 flask. Add sufficient acidified sodium sulfate to each extract so that some remains free-flowing. Stopper and allow the extracts to remain in contact with the sodium sulfate at least 4-hours or refrigerate overnight.

9.2.9 Concentration - Concentrate the extract by a Kuderna-Danish apparatus and N-Evap. Transfer the ether extract into the receiver through a funnel plugged with acid washed glass wool and topped with acidified sodium sulfate. Use liberal washings with ether to complete the transfer. Use a glass rod to crush any caked sodium sulfate and rinse with ether into the receiver. Concentrate the extract to approx. 3-ml.

9.2.10 Methylation – The methyl ester scan be formed with diazomethane by adding a diazomethane solution. Add 0.1-ml of MeOH to each extract and dilute with 1-ml of ether. Slowly add 2-mls of diazomethane solution to each extract. Let the extract stand for 10-minutes with occasional swirling. The yellow color must persist during this 10-minute process.

9.2.11 Solvent exchange – N-Evap the extract to 1-ml using a gentle stream of nitrogen. Add 1-ml of hexane and continue to nitrogen blow down. Do not let the extract go dry. Continue this process three more times. The yellow color will disappear during the blow-down. Add 0.1-grams of silicic acid to the extract. Dilute the extract to a final volume of 5-ml with hexane.

9.3 Extraction of waste samples.

9.3.1 If the waste sample is miscible with ether, weigh 1 to 2-grams into a 250-ml flask with 24/40 ground glass necks. Add all spiking solutions accordingly.

9.3.2 If the waste sample is not miscible with ether, try various solvents that are also miscible with ether to dissolve the sample. After the correct solvent combination is found, weigh 1 to 2-grams into a 250-ml flask with 24/40 ground glass necks. Add all spiking solutions accordingly.

9.3.3 Add 100-mls of ether to the flask.

9.3.4 Proceed with sections 9.2.6, 9.2.7, 9.2.8, 9.2.9, 9.2.10 and 9.2.11.

9.4 Instrument Calibration and Analysis

9.4.1 At the initiation of this method and thereafter, whenever continuing calibration fail criteria or when equipment changes are made (such as a new detector or new column is installed), establish a 5 point initial calibration (ICAL) standard curve. Additional ICAL standards may be analyzed for additional options for generating valid ICALs for each compound.

9.4.2 Prepare a 5-point curve (ICAL) by injecting the calibration standards described in Section 5.12. Spike 10-uls of the internal standard (DBOB) into each 1- ml calibration solution prior to injection into the GC/ECD. Calculate response factors (RF, see equations below) from the areas for the internal standard and the analyte peaks for each compound at each concentration level. Calculate the average response factor (RF_m) and the associated standard deviation (SD) from the five RFs obtained for five concentration levels.

9.4.3 Linearity – If the % RSD of any compound is 20% or less, then the relative response factor is assumed to be constant over the calibration range and the average relative response factor may be used for quantitation.

9.4.4 If the % RSD of any detected compound is greater then 20%, use least square regression methods to calculate sample concentrations. Non-linear regression procedures may require more than 5 points (minimum for ICAL). For regression methods, the instrument response is treated as the dependent variable (y) and the standard amount is the independent variable (x). When "Cs" is used as "x", use "As*Cis/Ais" as "y". When "Cs/Cis" is used as "x", use "As/Ais" as "y". Do not include origin (0,0) as an extra calibration point.

9.4.5 Weighted least squares method is preferred for calculating concentrations more accurately at lower levels. A linear least squares equation (y = a*x + b) is preferred over non-linear calibration equations (y = a*x2 + b*x + c). The coefficient of determination (COD), coefficient of variation (r2) or correlation coefficient (r) should be used to measure the goodness of fit. For regression models to be used for quantitative purposes, the COD, r2 or r must be greater than or equal to 0.99. A value of 1.00 indicates a perfect fit. Corrective action may be required if the criteria for %RSD, r, r2, or COD are not met.

9.4.6 Document ICAL in Form-6 equivalents, or in linear regression charts (See Appendix 2 for details). Use the following calculations for determining response factors and determining %RSD:

$$RF = (A_s C_{is}) / (A_{is} C_s)$$

where:
A_s = Area of the compound being measured
A_{is} = Area of the specific internal standard
C_{is} = Concentration of the specific internal standard
C_s = Concentration of the compound being measured

$$\% RSD = ((SD)/(RF_m))*100\%$$

where:

RF_m = mean of the 5 initial RFs for a compound
SD = standard deviation of the 5 initial 5 RFs for a compound.

Where SD is the square root of the following equation:

$$(SD^{**}2) = \frac{\sum_{i=1}^{n} (RF_i - RF_m)}{(n-1)}$$

n = number of levels of standards

9.4.7 An initial calibration (ICAL) should be considered a single event process. Consult the QA office or check applicable EPA methods (e.g. SW-846 Method 8000C and 8081A) for additional guidance on appropriate corrective action.

9.4.8 Re-fitting of ICAL – The selected model for ICAL may be subjected to an additional re-fitting check to establish representativeness of the model for each compound in each ICAL standard. This is an optional procedure, which is recommended but not required since automated data processing procedures may not be able to accommodate it. Calculate the %D between the calculated amount (Cc) for each compound in each ICAL standard from the ICAL model and its expected value (Ce). Ideally, the %D ((Cc-Ce) * 100 /Ce) for each compound in ICAL standard should meet the QC criteria for %RSD. It is possible for one or more ICAL standards to have %D exceeding the %RSD criteria, while the %RSD for ICAL model is within QC limits. The re-fitting procedure may be used to identify ICAL standards that need re-analysis or dropped from the calculation of ICAL model when re-analysis was not performed.

9.4.9 Check and optimize instrument operating conditions including automated peak integration procedures for appropriateness in addressing the chromatographic needs of the specific compound with problems.

9.4.10 If the problem appears to be associated with just one of the ICAL standards, that one standard may be reanalyzed once immediately (must be within 8-hours) after the ICAL. Discard all the results from the original analysis of the standard in question and replace it with the results from re-analysis.

9.4.11 When additional calibration standards (six or seven instead of five) are analyzed, narrowing the range of concentrations to be used for determining the goodness of fit is an acceptable procedure. Five or more standards should remain for the calculation of RSD or linear regression for each compound after one or more points are removed from the ICAL. More points (6 for second order and 7 for third order) may be needed for non-linear regression. It is generally prohibited to remove data points from within a calibration range, while still retaining the extreme ends of the calibration range. Do not discard a middle point unless it appears to have an obvious problem and six or more standards were analyzed for

ICAL and re-analysis of the affected standard was not performed within the required time limits. Changes to the upper end of the calibration range will affect the need to dilute samples above the range. A change to the lower range is not recommended since it will affect the method quantitation limit (MQL) used to report data. The default MQLs, which are based on the lowest ICAL concentration are stored in the LIMS and project-specific changes are not practical.

9.4.12 The refitting procedures may identify failed %Ds for upper level calibration standards for average response factor methods. The analyst may take alternate corrective actions such as the analysis of sample extracts under dilution for the affected compounds.

9.4.13 The refitting procedures may lead to failed %Ds for lower level calibration standards for regression methods. The analyst may take appropriate corrective action such as flagging the sample result as an estimated amount ("J" flag) at the lower level encountered in the sample.

9.4.14 Grand mean RSD – Previous versions of the EPA method(s) had provisions for the calculation of the grand mean %RSD for all target compounds in the ICAL solution, when the % RSD of any detected compound is greater than 20%. The grand mean %RSD should be <20% for ICAL to be acceptable. This procedure is discontinued. Criteria for Initial calibration are compound-specific.

9.4.15 The term "calibration verification" (CALV) is used to describe initial verification (ICV) of the initial calibration (ICAL) using second-source standards and subsequent continuing verifications (CCVs) using same-source standards. The response factors calculated during CALV are not used for sample quantitation since CALV standards are not used as "continuing calibration" standards permitted in some of the EPA methods.

9.4.16 Initial Calibration Verification (ICV) - Analysis of a second source ICV standard is required before any samples can be analyzed for USACE projects. Since second source standards are not employed for the analysis of LCS/LCSD and MS/MSD samples, ICV analysis is applicable for all projects. Calculate the percent difference (%D) or %Drift for the observed response factor in ICV from the mean RF as described for CCV. The %D or %drift should not exceed □15% for each target compound. Document calibration verification in instrument printouts as shown for CCVs. Check the ICAL and ICV standards if it is not achievable for all the target compounds without any exception. Use professional judgment for non-USACE projects and for compounds that are not contaminants of concern (COC) for USACE projects. The ICV may be used as the beginning CCV for sample analysis.

9.4.17 After the initial calibration curve has been established and verified with an ICV, analytical sample may be analyzed. A continuing calibration check (CCV) can be made, by injecting the mid-point calibration solution (as described in Section 5.12.3) after every 10 injections. The percent difference (%D) for the RF from the continuing calibration check must be within 15% of the mean RF (RF_m) obtained during the initial calibration

(ICAL). Document calibration verification in Form-7 equivalents (See Appendix 3 for details). The % difference is calculated as follows:

$$\% D = ((RF_v - RF_m) * 100) / RF_m$$

where:

RF_m = Average RF for the compound from ICAL
RF_v = Measured RF for the compound in CALV (ICV or CCV).

9.4.18 If the %D for any compound is greater than 15%, the GC should be restored to proper operating condition before re-analyzing the CCV for all the compounds. A new ICAL should be generated if the CCV does not meet QC criteria.

9.4.19 Grand mean %D – Previous versions of the EPA method(s) had provisions for the calculation of the grand mean %D for all target compounds in the CCV standard, when the %D of any detected compound is greater than 15%. The grand mean %D should be <15% for CCV to be acceptable. This procedure is discontinued. Criteria for CCV are compound-specific.

9.4.20 Calibration Factors are used when external standards are used for calibration. This procedure is not used at AML and they are described only when they are necessary. Situations include internal standard coeluting with one of the target compounds in one or more columns. When external standard calibration is employed, calculate the calibration factor for each analyte at each concentration, the mean calibration factor, and the relative standard deviation (RSD) of the calibration factors. If external standard calibration is employed, refer to Method 8000C or AML SOP on organochlorine pesticides or PCBs (8081 0r 8082) for the calculation of calibration factors.

9.4.21 Retention time windows are established by averaging the retention times, preferably over a 3-day period. Center the window around the daily calibration standard. Continuing calibration standards retention times may shift not more than twice the recovery window for the run to be considered valid. Any shift in retention times must be considered when evaluating sample results. Determine and document retention time window for each compound in logbooks (See Appendix 4 for details). Wider windows may be used to minimize false negatives. However, the window should not be too wide since false positives may result.

10.0 DATA ANALYSIS AND CALCULATIONS

10.1 The internal standard technique is used to determine the concentration of an identified target compound. For water samples the calculation is as follows:

$$\text{Concentration } (\mu g/L) = \frac{(As) * (IS) * (Ve)}{}$$

(Ais) (RFm) (Vs)

where:

As = Area for compound being measured.
IS = Concentration of the internal standard in the extract injected (µg/mL)
Ais = Area for the internal standard
RFm = Mean Response factor for compound being measured
Vs = Volume of water (L) extracted
Ve = Volume of the final extract (mL), taking into account any dilutions made

10.2 For soil samples the calculation is as follows:

$$\text{Concentration } (\mu g/kg) = \frac{(As) * (IS) * (Ve) * (100)}{(Ais) * (RFm) * (Ws) * (\%S)}$$

where:

As = Area for compound being measured.
IS = Concentration of the internal standard in the extract injected (µg/mL)
Ais = Area for the internal standard
RFm = Mean Response factor for compound being measured
Ws = Wet weight of soil sample (kg) extracted
Ve = Volume of the final extract (mL), taking into account any dilutions made

10.3 Calculate the matrix spike (MS) and/or MS duplicate (MSD) recoveries for each analyte from the following formula:

$$\text{Recovery} = \%R = \frac{Cs - Cu}{Ct} * 100$$

Where
Cs = Measured concentration of the analytes in the spiked sample
Cu = Measured concentration of the analytes in the unspiked sample
Ct = True value or spike concentration of the analytes

10.4 Calculate the laboratory control sample (LCS) and/or LCS duplicate (LCSD) recoveries for each analyte using the following equation:

$$\text{Recovery} = \%R = \frac{Cs}{Ct} * 100$$

Where

Cs = Measured concentration of the analytes in the LCS/LCSD
Ct = True value (spike amount) concentration of the analytes in the LCS/LCSD

10.5 Precision can be estimated from the relative percent difference (RPD) of the two sample results obtained for the spiked samples (MS & MSD and LCS and LCSD). Calculate the relative percent difference from the following formula:

$$RPD = \frac{|R1 - R2|}{\{R1+R2\}/2} * 100$$

where
R1 = Measured sample result of the first sample aliquot
R2 = Measured sample result of the second sample aliquot

10.6 Surrogate – Spike every sample and QC sample in an analytical QC batch. Calculate surrogate recoveries using the following formula:

$$\%Recovery = \frac{\text{conc. of surrogate in the sample}}{\text{true value for the conc. of the surrogate}} * 100$$

11.0 QUALITY CONTROL

11.1 Sample preparation logs, sample data and quality control data should be gathered prior to review. Checklists for documenting raw data and their verification (See Appendix 5 for details) should be used to document verification of calculations and adherence to method procedures. Calculated sample results for each compound should be compared to their method detection limits (MDL), lowest level for reporting (LLR), reporting limits (RL) and method quantitation limits (MQL) as shown in Tables IX and X. Exceptions should be documented. Samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on these checklists. Summarize sample results using Form-1 equivalents (See Appendix 7 for details). Summarize QC results in Form-1 equivalents and recoveries and RPDs using Form-3 equivalents for MS/MSD (See Appendix 9 for details) and LCS/LCSD (See Appendix 10 for details). Use data verification and validation procedures described in detail in Section 12 for appropriate corrective action that may be required if QC elements are not in control.

11.2 Surrogate recovery should be within QC limits for samples, spikes and blanks. Document recoveries using Form-2 equivalents (See Appendix 11 for details). The laboratory currently uses QC limits from the Department of Defense Quality Systems Manual (DoD QSM, Final Version 2, June 2002) for surrogate recoveries. Flag recoveries that are outside the QC limits with an asterisk (*). If re-extraction and/or

reanalysis have been performed, and the surrogate recoveries for reanalysis are within QC limits, report data based on acceptable surrogate recoveries. If the surrogate recoveries are outside the QC limits for the reanalysis, report data for both analyses with surrogate recovery flags.

11.3 Method blanks (MB) should be free of target compounds. Report target compounds that are present in the MB at levels greater than the reporting limit (RL) using J qualifiers if the levels are less than method quantitation limits (MQL). The action level for method blank is one half of the MQL. See tables IX and X for a list of MDLs, RLs, and MQLs. Document method blank or QC association using Form-4 equivalents (See Appendix 8 for details). Samples associated with the method blank containing target compounds at levels greater than on half the MQL will require re-extraction and reanalysis unless other criteria acceptable to the client are used. Since method blanks may be contaminated when they are associated with samples containing high levels of target compounds, only samples that contain less than one tenth of the MB amount may need to be re-extracted and reanalyzed.

11.4 LCS recoveries should be within QC limits (DoD QSM) for the target compounds. Flag recoveries that are outside the control limits with “ME” and compounds outside the ME limits with “ME*”. See Tables IX and X for LCS recoveries and QC limits. Contact supervisor for appropriate corrective action if more than the allowed numbers of compounds are outside the control limits or the marginal exccedance (ME) limits. Data qualification, re-extraction, and/or reanalysis of associated samples may be necessary depending on project requirements, turnaround time and other constraints.

11.5 MS recoveries should be within QC limits (DoD QSM) for the target compounds. Flag recoveries that are outside the control limits with “ME” and compounds outside the ME limits with “ME*”. See Tables IX and X for spike recoveries and QC limits. Contact supervisor for appropriate corrective action if more than 10% of the compounds are outside the QC limits. Data qualification, re-extraction, and/or reanalysis of associated samples may be necessary depending on project requirements, turnaround time and other constraints.

11.6 Report target compounds that are present at levels greater than the method detection limit (MDL), lowest level for reporting (LLR) or the reporting limit (RL) as appropriate (See Project Plan for details) using "J" qualifiers if the levels are less than MQL. Compounds that were detected in the associated MB should also be qualified with a "B" code. If the concentration of any of the target compounds exceed the upper limit of calibration (ICAL), flag the affected compounds data with an estimated or "E" flag. The extracts may need to be diluted and reanalyzed until the concentrations are within the calibration limits for normal turnaround analyses. For rapid turnaround analyses sample results may be reported with "E" qualifiers if sufficient time is not available for reanalysis. Prepare Form-1 equivalents (See Appendix 7) for sample reports.

12 **DATA VERIFICATION AND VALIDATION -** This section will provide guidance and procedures to assess if data produced using this SOP are valid and legally defensible. Document review and comments on the checklist (See Appendix 5). Verify that sample preparation logs are complete and the instrument run logs are available for review (See Appendix 8). Anomalies and exceptions should be documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run

under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on the checklists. Army (USACE) and Air Force (AFCEE) projects may have special requirements (See Appendix 13) that will require extra attention. A simple, universal procedure for evaluating and choosing appropriate corrective action doe not exist to deal with situations when one or more QC parameters are outside the acceptance window. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints.

12.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) projects may have additional requirements that are related to USACE laboratory validation (CX), USACE District requirements, government contract requirements, specific USACE project requirements or USACE Architect/Engineer (A/E) requirements. USACE-CX requirements (Summary of Method Quality Objectives for Method 8151), AFCEE requirements and the DoD QSM requirements are included in Appendix 13.

12.1.1 The USACE requirements shown in Appendix 13 and elsewhere in this SOP are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may or may not be attainable for routine sample analysis. Since USACE MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

12.1.2 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality objectives and/or other QC requirements. Exceptions and data qualifications necessary should be documented on the checklists and eventually on case narratives.

12.1.3 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist, perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete. For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete. For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director will review the checklists (initial/date as necessary), prepare case

narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

12.1.4 The blank criteria shown in Appendix 13 has been modified by USACE auditor(s) as one half of the applicable MQL (personal communications, USACE auditor, 1999).

12.2HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

12.2.1 WATER - The holding times for sample extraction is 7 days from sampling or from the date of TCLP leachate preparation.

12.2.2 SOIL – A 14 day holding is used for soil and waste samples.

12.2.3 TCLP – TCLP leachates should be prepared within 14-days of waste sample collection. TCLP leachates should be extracted within 7 days after TCLP leachate preparation.

12.2.4 Sample extracts should be analyzed within 40-days from the date of sample extraction. This applies to extracts from aqueous, soil and leachate samples.

12.2.5 Extraction holding times are established by comparing the sampling dates on the chain of custody with the date of sample preparation. TCLP holding times are established by comparing first the COC to the leaching date and then the leaching date to the sample preparation date. Analysis holding times are calculated by comparing the extract injection dates for the instrumental analysis with the sample extraction dates on the sample preparation log.

12.2.6 ACTION – If holding times have been exceeded, contact the client. All positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or invalid (I).

12.3 INITIAL CALIBRATION – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the GC/ECD is capable of producing data within a range that is linear. The ICAL should be verified (ICV) using a second-source standard for USACE and AFCEE projects (See Appendix 13 for details).

12.3.1 Compounds – Check to see if the mean RF for any compounds is outside the criteria of 20% or less for %RSD. If the %RSD for a compound is outside the 20% criteria, check to see if the mean RSD for all the target compounds is less than 20%. If mean RSD is less than 20%, check to see if linear regression can be used for compounds with %RSD greater than 20%. The limit for the correlation coefficient (r) used in linear regression is 0.990.

12.3.2 ACTION – Positive results for compounds not meeting the criteria may need to be qualified as estimated (J) by the user (client). A new calibration may need to be constructed if a significant number of compounds need qualification. Check the standard solutions and the instrument for problems, correct any identified problem(s) and repeat initial calibration, if necessary. For compounds with QC

problems, report detects and non-detects without qualifiers and mention the outliers in the case narrative.

12.4 INITIAL CALIBRATION (ICAL)– Instrument calibration (See Appendix 3 for details) criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the GC/ECD is capable of producing data within a range that is linear. The ICAL includes initial verification (ICV) of the ICAL with a second source standard for USACE and AFCEE projects (See Appendix 13 for details).

12.4.1 CHECK – Check to see if any other compounds are outside the criteria of 20% or less for %RSD. If a compound is outside the 20% criteria, check to see if regression methods are used. The goodness of fit should be measured with the coefficient of determination (COD) or related quantities (r2 and r) greater than 0.99.

12.4.2 ACTION – Check and correct the standards, including the range of concentrations for appropriateness and repeat the ICAL analysis.

12.4.3 ACTION - Report the problem to the Project Manager and report detects and non-detects with appropriate qualifiers for rapid TAT projects. Mention the ICAL anomaly in the case narrative.

12.5INITIAL CALIBRATION VERIFICATION – Calibration verification includes initial calibration verification (ICV) and continuing calibration verifications (CCVs). A second source ICV is used to check the integrity of the ICAL standards used for calibrating the instrument. The 12-hour analytical shift begins with the injection of the ICV (instead of a CCV) for sample analyses that are performed immediately after an ICAL.

12.5.1 Target Compounds - Check to see if %D for any of the target compounds is outside the USACE QC criteria (<±15%).

12.5.2 ACTION - Check the ICAL and ICV standards for integrity. Replace one or more of the working standards (ICV first and ICAL later) for trouble shooting. Correct the problem(s) and repeat ICV analysis. Use professional judgment for non-USACE projects.

12.5.3 ACTION - When ICAL and ICV are acceptable based on SW-846 criteria, positive results and non-detects for target compounds not meeting the ±15% %D may be reported without data qualifications for non-USACE projects provided a valid CCV was obtained immediately following the CCV. The ICAL and/or ICV standards should be inspected and replaced, if necessary for compliance with this requirement for USACE projects.

12.6CONTINUING CALIBRATION VERIFICATION – The continuing calibration verification (See Appendix 4 for details) is used to check that the instrument is still operating under the linear range of the initial calibration and has remained stable every 12 hours.

12.6.1 IS area counts (use mean IS area from ICAL for the %D of IS area) must not vary by more than a factor of 2 (-50% or + 100%) from the associated ICAL. Check to see if the %D for any compounds is outside the criteria of 15% or less.

12.6.2 %D for Compounds present in the sample – The %D for the CCVs before and after the sample analysis should be within QC limits when internal standards are

not used. The %D for the CCV before sample analysis should be within QC limits when internal standards are used.

12.6.3 The %D for Compounds not present in the sample – The %D for the CCVs before and after the sample analysis may be >(+15%). Corrective action is not required unless the %D is < (-15%).

12.6.4 ACTION – Corrective action must be performed for positive results with %D outliers. In exceptional cases (e.g. rapid TAT analysis for screening level data), positive results for compounds not meeting the criteria may be qualified as estimated (J) by the user (client).

12.6.5 ACTION - Report non-detects without qualifiers and include a description of the anomaly in the case narrative.

12.6.6 ACTION – Perform more frequent CCVs (e.g. every 10 samples) than required (every 12-hours) to avoid extensive re-analysis.

12.7 METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Sample data qualification to alert blank contamination is performed by the laboratory. Sample data qualification based on all the blank results (laboratory MB, equipment blank, and other field blanks) is performed by the user (client). The processing of blanks helps eliminate reporting of false positives in project reports.

12.7.1 Method blanks should be free of all target analytes and other interfering non-target analytes. To be acceptable, blank results should not exceed one half of the applicable MQL for any compound.

12.7.2 Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples. If levels greater than one half the MQLs are present, inspect and correct the problem. Reanalyze all associated samples. Contact client if other criteria are applicable. The only exception may be quick turn around projects.

12.7.3 ACTION – All positive results are reported with a “B” code, if contaminants are found in the method blank.

12.7.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, then no action is taken.

12.7.5 ACTION – Any compound detected in the sample which were also detected in associated method blank at levels greater than one half the MQLs should be re-extracted unless other client criteria are applicable.

12.7.6 ACTION – If contaminants resulting in GC/ECD peak saturation are present, all affected compounds are qualified as invalid (I).

12.8 SURROGATE RECOVERIES – Surrogates or system monitoring compounds are different structurally, but similar chemically to classes of target compounds. These surrogate compounds are added just prior to extraction of the samples and the percent recoveries of the surrogates are calculated and evaluated.

12.8.1 Verify that the surrogate recoveries (See Table IX and X) for the surrogate compound is within the DoD QSM control limits.

12.8.2 ACTION – When surrogate recoveries are outside control limits the sample must be re-analyzed. Reanalysis with or without cleanup may be performed under dilution, if necessary if original analysis should indicate high levels of target and non-target analytes.

12.8.3 ACTION - Reextraction and cleanup will be necessary if reanalysis does not resolve the problem. If surrogate recoveries are still outside the limits, report both sets of data and describe the problem in the case narrative.

12.9 LABORATORY CONTROL STANDARD (LCS) – The analysis of LCS samples is used to evaluate method competence in the absence of matrix effects. The LCS is evaluated by comparing the percent recovery for all of the target analytes to the laboratory QC limits, which are identical to the 3-sigma DoD QSM QC limits. See Appendix 10 for guidance on documenting LCS and LCSD concentrations and %R in QC summary report forms. The DoD QSM specifies the number of compounds that can exceed the 3-sigma QC limits based on statistical considerations. The LCS recovery for such compounds should not exceed the marginal exceedance 4-sigma (ME) limits. Evaluation procedures may include comparison of LCS recoveries with those determined by project-specific DQOs (See project QAPP, if available), laboratory statistical control limits, or USEPA guidance. Initially, the effect of QC failure on the samples should be evaluated. Regardless of this assessment, steps shall be taken to find the source of the problem and correct it.

12.9.1 CHECK - LCS and LCSD recoveries (See Appendix 8 for details) should be within QC limits (See Tables IX and X). Compare the %R results for each compound in LCS with QC limits (control and marginal exceedance) to see if all the results are within the acceptable range. Flag outliers with “ME” if it is within the 3-sigma limits. Flag it with “ME*” if it is outside the 4-sigma ME limits.

12.9.2 CHECK - Compare observed %R results for each compound with the laboratory limits for LCS/LCSD recoveries (e.g. USACE criteria). Perform manual calculation for %R, if necessary for QC outliers. Document the number of compounds that exceeded the QC limit and compare it against the maximum recommended by DoD QSM. Document this review in checklists and in case narratives.

12.9.3 ACTION - Typically, the LCS would be reanalyzed for the failed analytes (ME*) only. If the second analysis fails, then the LCS, method blank, and all associated samples of the batch would be reanalyzed for the failed analytes only. If sufficient sample is not available for reanalysis, or if the corrective action is ineffective, discuss the situation with the client project manager (or chemist). Include a discussion of the situation in the case narrative. The case narrative should discuss the corrective action taken and any other information.

12.9.4 ACTION - For non-USACE projects, no action may need to be taken on LCS and/or LCSD recovery data alone either to reanalyze samples and/or to qualify associated samples. Providing data of known quality is sufficient for most projects. If significant deviations (e.g. number of target compound exceeding QC limits for recoveries is greater than the allowable maximum) are noticed, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements, which may be compound specific. Check with

the project manager about such requirements. Professional judgment may be used by the user (client) in evaluating sample data qualification options in conjunction with other QC data for the project.

12.10 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike recovery data are used to evaluate the accuracy of the analytical method on project samples. The evaluation is performed by comparing the %R for target analytes to the DoD QSM limits established for the LCS samples.

12.10.1 Document MS/MSD recoveries in instrument printouts and in LIMS generated reports as shown in Appendix IX. Flag QC outliers with "ME" and "ME*" on client reports. The MS/MSD percent recoveries should be within QC limits (Tables IX and X). Perform one manual calculation using raw data and extraction sheet information.

12.10.2 MS data evaluation is more complex than method blank or LCS data evaluation since they are complicated by matrix effects in addition to sample preparation and analysis errors. The heterogeneity of grab soil samples, and potential non-representativeness of sequentially collected water samples, etc. further complicate the evaluation since it is assumed that the native concentrations are constant in triplicate analyses. In addition, concentrations of the target analytes in the sample can also far exceed spike amounts. If the native concentration of target analytes in the sample chosen for spiking is high relative to the spiking concentration, the differences in the native concentration between the unspiked sample and the spiked samples may also become very significant.

12.10.3 In addition to their reluctance to pay for MS and/or MSD analyses, most clients may not know what samples to collect for MS analysis. Clients may not collect sufficient samples, have no knowledge of the native analyte concentrations in the chosen sample, and may not have knowledge of MS spiking levels that are appropriate either for the sample or their project. For procedures with multiple target analytes, spiking levels may be inappropriate for one or more analytes, if samples that contain target analytes are chosen either in the field or by the laboratory. If no project samples are collected for MS analysis, samples from another project used for MS analysis may not be representative of the project samples. When project samples (e.g. surface soil) are chosen, their MS recovery characteristics may be different from those of other samples (e.g. sub-surface soil) in the same batch.

12.10.4 ACTION - If the sample chosen for MS/MSD is inappropriate, notify the client. When additional payments are authorized for re-analysis, re-extract and/or reanalyze the MS/MSD samples after employing cleanup procedures (if applicable), dilution techniques to minimize matrix interference, or using other project samples that appear to be clean, when sufficient quantities are available. Re-spike the sample at a higher level (e.g., at approximately two to four times the sample concentration), then reanalyze the sample based on project-specific requirements. It is the responsibility of the client at all times to select appropriate samples for MS analysis, make payments for additional analyses, and to offer necessary guidance as needed to meet their data quality objectives.

12.10.5 ACTION - If the matrix effect cannot be resolved, discuss the situation in the case narrative. No action may be taken by the laboratory to qualify associated sample data based on MS/MSD recovery data alone. Professional judgment may be used by the user (client) in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

12.10.6 ACTION – No action is taken on MS/MSD recoveries data alone to qualify all associated samples. Since matrix interference could lead to lead to serious analytical problems, the supervisor should be consulted when significant outliers are noted. Document QC outliers for %recovery and RPD on the checklist and on client reports and draw attention to the QC outliers on the case narrative. No action may be taken on MS/MSD recovery data alone either to reanalyze samples and/or to qualify associated samples either by the analyst or by the end user. Professional judgment may be used by the user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

12.11 INTERNAL STANDARD PERFORMANCE – Internal Standard (IS) performance criteria ensures that the GC sensitivity and response is stable during each run.

12.11.1 IS area counts (use mean IS area from ICAL for the %D of IS area) must not vary by more than a factor of 2 (-50% or + 100%) from the associated ICAL/Calibration Verification standard. The internal standard areas for the CCV should not differ by a factor of 2 (-50% or + 100%) from the initial calibration of the same standard.

12.11.2 The retention time of the internal standard must not vary by more than 30 seconds from the associated daily calibration standard.

12.11.3 ACTION – If an internal standard is outside of QC limits of the associated daily standard, then re-analysis is required or a dilution may be required if it appears that interference's occur from target and non-target analytes at high concentrations relative to the ICAL. Do not dilute the sample to the point where all target analytes are non-detect. Professional judgment must be used when dilutions are required for IS criteria.

12.11.4 ACTION – If an internal standard is outside of QC limits of the associated ICAL or CCV standard, correct the problem and re-analyze samples with a valid CCV. If re-analysis has been performed and it is still out, it is of the professional judgment of the analyst to dilute or not to dilute the sample further so as to not to dilute out any target analytes.

12.11.5 ACTION - Report results without qualification describing the QC problem in the case narrative. The user (client) may qualify positive results for compounds quantitated using the affected IS as estimated (J). If a very low IS area is reported, indicating a loss of sensitivity, non-detects may require qualification as invalid (I) or rejected (R). Document recommendation for the client in the case narrative.

12.11.6 ACTION – If an IS retention time varies by more than +/- 30 seconds, the total ion profile must be checked for any false positives or negatives. If a large shift

has occurred, then professional judgment must be used as to whether reanalyze or not. See previous section for guidance.

12.12 TARGET COMPOUND IDENTIFICATION – Criteria for GC/ECD qualitative analysis is to minimize the number of erroneous positive or negative identifications of compounds.

12.12.1 Positive compounds must be within +/- 0.06 relative time (RRT) units of the standard RRT. The results should be >MDL or other threshold for reporting positives (e.g. LLR, RL) on both columns.

12.12.2 The RPD for sample amounts from the two columns for identified compounds should be within <40%. Document RPD calculations (See Appendix 6 for details) in Sample Result worksheets (Form-10 equivalents).

12.12.3 ACTION - If the RPD for positive results are <40%, report the result from the primary column without any flag.

12.12.4 ACTION - If the RPD is >40%, report the result from the primary column with a "J" flag. The result from the confirmation column may be reported with a "J" flag at the discretion of the analyst, if interference is suspected for the compound of interest in the primary column.

12.12.5 Professional judgment must be used when applying the criteria for GC/ECD analysis of target analytes. Take into account factors such as matrix effect and column bleed.

13 REFERENCES

13.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8151A, Chlorinated Herbicides by GC using Methylation or Pentafluorobenzylation Derivatization, December 1996.

13.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.

13.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

13.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3500B, Organic Extraction and Sample Preparation, December 1996.

13.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3600C, Cleanup, December 1996.

13.6 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3640A, Gel-Permeation Cleanup, December 1996.

13.7 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3660B, Sulfur Cleanup, December 1996.

13.8 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3580A, Waste Dilution, December 1996.

13.9 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.

13.10 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

13.11 AML Standard Operating Procedures, Q-Water-RC.

13.12 AML Standard Operating Procedures, Q-WasteMgmt-RD.

13.13 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

13.14 AML Standard Operating Procedures, Q-Controlcharts-RC.

Table I

HERBICIDE CALIBRATION STOCK SOLUTION

Compound	Stock Solution Concentration (ug/ml)
2,4-D	100
Dalapon	250
2,4-DB	100
Dicamba	10
Dichlorprop	100
Dinoseb	50
MCPA	10000
MCPP	10000
Silvex (2,4,5-TP)	10
2,4,5-T	10

NOTE: The stock solution is used for calibration.

Table II

HERBICIDE CALIBRATION INTERMEDIATE SOLUTION
and
HERBICIDE INITIAL CALIBRATION SOLUTIONS (HERB-5)

Compound	Intermediate Solution (also Herbicide STD-5) Concentration (ng/ml)
2,4-D	1000
Dalapon	2500
2,4-DB	1000
Dicamba	100
Dichlorprop	1000
Dinoseb	500
MCPA	100000
MCPP	100000
Silvex (2,4,5-TP)	100
2,4,5-T	100

NOTE: The intermediate solution also serves as the high point for the initial calibration (ICAL-HIGH).

Table III

HERBICIDE INITIAL CALIBRATION SOLUTIONS (HERB-4)

Compound	Intermediate Solution Concentration	Intermediate Solution Volume	Hexane Volume	Herbicide STD-4 Concentration
2,4-D	1000 ng/ml	700 uls	300 uls	700 ng/ml
Dalapon	2500 ng/ml	700 uls	300 uls	1750 ng/ml
2,4-DB	1000 ng/ml	700 uls	300 uls	700 ng/ml
Dicamba	100 ng/ml	700 uls	300 uls	70 ng/ml
Dichlorprop	1000 ng/ml	700 uls	300 uls	700 ng/ml
Dinoseb	500 ng/ml	700 uls	300 uls	350 ng/ml
MCPA	10000 ng/ml	700 uls	300 uls	7000 ng/ml
MCPP	10000 ng/ml	700 uls	300 uls	7000 ng/ml
Silvex	100 ng/ml	700 uls	300 uls	70 ng/ml
2,4,5-T	100 ng/ml	700 uls	300 uls	70 ng/ml

NOTE: This solution serves as the mid-high point for the initial calibration (ICAL-MID HIGH).

Table IV

HERBICIDE INITIAL CALIBRATION SOLUTIONS (HERB-3)

Compound	Intermediate Solution Concentration	Intermediate Solution Volume	Hexane Volume	Herbicide STD-3 Concentration
2,4-D	1000 ng/ml	500 uls	500 uls	500 ng/ml
Dalapon	2500 ng/ml	500 uls	500 uls	2250 ng/ml
2,4-DB	1000 ng/ml	500 uls	500 uls	500 ng/ml
Dicamba	100 ng/ml	500 uls	500 uls	50 ng/ml
Dichlorprop	1000 ng/ml	500 uls	500 uls	500 ng/ml
Dinoseb	500 ng/ml	500 uls	500 uls	250 ng/ml
MCPA	10000 ng/ml	500 uls	500 uls	5000 ng/ml
MCPP	10000 ng/ml	500 uls	500 uls	5000 ng/ml
Silvex	100 ng/ml	500 uls	500 uls	50 ng/ml
2,4,5-T	100 ng/ml	500 uls	500 uls	50 ng/ml

NOTE: This solution serves as the mid point for the initial calibration (ICAL-MID). This solution also serves as the calibration verification standard.

Table V

HERBICIDE INITIAL CALIBRATION SOLUTIONS (HERB-2)

Compound	Intermediate Solution Concentration	Intermediate Solution Volume	Hexane Volume	Herbicide STD-2 Concentration
2,4-D	1000 ng/ml	300 uls	700 uls	300 ng/ml
Dalapon	2500 ng/ml	300 uls	700 uls	750 ng/ml
2,4-DB	1000 ng/ml	300 uls	700 uls	300 ng/ml
Dicamba	100 ng/ml	300 uls	700 uls	30 ng/ml
Dichlorprop	1000 ng/ml	300 uls	700 uls	300 ng/ml
Dinoseb	500 ng/ml	300 uls	700 uls	150 ng/ml
MCPA	10000 ng/ml	300 uls	700 uls	3000 ng/ml
MCPP	10000 ng/ml	300 uls	700 uls	3000 ng/ml
Silvex	100 ng/ml	300 uls	700 uls	30 ng/ml
2,4,5-T	100 ng/ml	300 uls	700 uls	30 ng/ml

NOTE: This solution serves as the low-mid point for the initial calibration (ICAL-LOW MID).

Table VI

HERBICIDE INITIAL CALIBRATION SOLUTIONS (HERB-1)

Compound	Intermediate Solution Concentration	Intermediate Solution Volume	Hexane Volume	Herbicide STD-1 Concentration
2,4-D	1000 ng/ml	100 uls	900 uls	100 ng/ml
Dalapon	2500 ng/ml	100 uls	900 uls	250 ng/ml
2,4-DB	1000 ng/ml	100 uls	900 uls	100 ng/ml
Dicamba	100 ng/ml	100 uls	900 uls	10 ng/ml
Dichlorprop	1000 ng/ml	100 uls	900 uls	100 ng/ml
Dinoseb	500 ng/ml	100 uls	900 uls	50 ng/ml
MCPA	10000 ng/ml	100 uls	900 uls	1000 ng/ml
MCPP	10000 ng/ml	100 uls	900 uls	1000 ng/ml
Silvex	100 ng/ml	100 uls	900 uls	10 ng/ml
2,4,5-T	100 ng/ml	100 uls	900 uls	10 ng/ml

NOTE: This solution serves as the low point for the initial calibration (ICAL-LOW).

Table VII

HERBICIDE MS/MSD SPIKING SOLUTIONS

Compound	MS/MSD Spike Soln. Concentration	MS/MSD Spike Soln. Volume (mL)	Aqueous Sample Volume(mL)	Final Sample Extract Volume (mL)	Conc. ng/mL
2,4-D	2500 ng/ml	1-mL	1000-mL	5-mL	500-ng/mL
Dalapon	11250 ng/ml	1-mL	1000-mL	5-mL	2250-ng/mL
2,4-DB	2500 ng/ml	1-mL	1000-mL	5-mL	500-ng/mL
Dicamba	250 ng/ml	1-mL	1000-mL	5-mL	50-ng/mL
Dichlorprop	2500 ng/ml	1-mL	1000-mL	5-mL	500-ng/mL
Dinoseb	1250 ng/ml	1-mL	1000-mL	5-mL	250-ng/mL
MCPA	25000 ng/ml	1-mL	1000-mL	5-mL	5000-ng/mL
MCPP	25000 ng/ml	1-mL	1000-mL	5-mL	5000-ng/mL
Silvex	250 ng/ml	1-mL	1000-mL	5-mL	50-ng/mL
2,4,5-T	250 ng/ml	1-mL	1000-mL	5-mL	50-ng/mL

Table VIII

HERBICIDE LCS/LCSD SPIKING SOLUTIONS

Compound	LCS/LCSD Spike Soln. Concentration	LCS/LCSD Spike Soln. Volume (mL)	Aqueous Sample Volume(mL)	Final Sample Extract Volume (mL)	Conc. ng/mL
2,4-D	2500 ng/ml	1-mL	1000-mL	5-mL	500-ng/mL
Dalapon	11250 ng/ml	1-mL	1000-mL	5-mL	2250-ng/mL
2,4-DB	2500 ng/ml	1-mL	1000-mL	5-mL	500-ng/mL
Dicamba	250 ng/ml	1-mL	1000-mL	5-mL	50-ng/mL
Dichlorprop	2500 ng/ml	1-mL	1000-mL	5-mL	500-ng/mL
Dinoseb	1250 ng/ml	1-mL	1000-mL	5-mL	250-ng/mL
MCPA	25000 ng/ml	1-mL	1000-mL	5-mL	5000-ng/mL
MCPP	25000 ng/ml	1-mL	1000-mL	5-mL	5000-ng/mL
Silvex	250 ng/ml	1-mL	1000-mL	5-mL	50-ng/mL
2,4,5-T	250 ng/ml	1-mL	1000-mL	5-mL	50-ng/mL

Table IX
Aqueous Sample MDL, LLR, MQL and QC Limits for LCS and MS

Chlorinated Herbicides Analysis													
Analytical Management Laboratories													
Preparation Method:	8151A						Derivatization reagent:				Diazomethane		
Analysis Method:	8151A						CAL standards:				Methyl esters		
Matrix: water	**MDL**	**MDL**	**MDL**	**MDL**	**LLR**	**MQL**	**Ratio (RQ)**		**DoD QSM LCS & MS Control Limits**		**DoD QSM LCS & MS ME Limits**		**RPD**
Compound	Spike	DB-5	DB-1701	Report	Report	Report	QL/DL	flag	Lower	Upper	Lower	Upper	Limit
	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L			**%R**	**%R**	**%R**	**%R**	%
Dalapon	0.375	0.066	0.039	**0.066**	0.132	**0.625**	9.5		40	110	40	110	50
Dicamba	0.015	0.021	0.031	**0.031**	0.061	**0.100**	3.3		60	110	60	110	50
MCPP	15	22.83	16.30	**22.83**	45.65	**100**	4.4		50	150	50	150	50
MCPA	15	5.02	17.04	**17.04**	34.08	**100**	5.9		60	145	60	145	50
Dichloroprop	0.15	0.042	0.075	**0.075**	0.15	**0.250**	3.3		70	120	70	120	50
2,4-D	0.15	0.035	0.052	**0.052**	0.103	**0.250**	4.8		35	115	35	115	50
Silvex (2,4,5-TP)	0.015	0.005	0.003	**0.005**	0.01	**0.025**	4.9		50	115	50	115	50
2,4,5-T	0.015	0.004	0.003	**0.004**	0.008	**0.025**	6.4		35	110	35	110	50
Dinoseb	0.075	0.015	0.006	**0.015**	0.031	**0.125**	8.1		20	95	20	95	50
2,4-DB	0.15	0.049	0.030	**0.049**	0.099	**0.250**	5.1		45	130	45	130	50
Surrogate:													
DCAA									50	150			
Criteria for the ratio (RD) of mean amount found to MDL: >1 but <5 for water and for MQL/MDL ratios (RQ) to be >3.													
Default QC limits (50% to 150%) are used for MCPP.													
RPD limits are those described for pesticides (SW 8081) in USACE EM200-1-3, Appendix I.													
Default AML QC limits are used for surrogate recoveries.													
The following criteria will apply for marginal exceedances:													
Number of allowed ME analytes per total analytes: 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).													
NOTE: Marginal exceedance concept is not applicable to herbicides analysis. Control limits and ME limits are identical.													
NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.													

Table X
Soil/sediment Sample MDL, LLR, MQL and QC Limits for LCS and MS

Chlorinated Herbicides Analysis

Analytical Management Laboratories

Preparation Method:	8151A						Derivatization reagent:				Diazomethane		
Analysis Method:	8151A						CAL standards:				Methyl esters		
Matrix: Soil	**MDL**	**MDL**	**MDL**	**MDL**	**LLR**	**MQL**	**Ratio (RQ)**		**DoD QSM LCS & MS Control Limits**		**DoD QSM LCS & MS ME Limits**		**RPD**
Compound	Spike	DB-5	DB-1701	Report	Report	Report	QL/DL	flag	Lower	Upper	Lower	Upper	Limit
	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg			**%R**	**%R**	**%R**	**%R**	%
Dalapon	12.5	2.62	0.90	**2.62**	5.23	**20.8**	8.0		40	110	40	110	50
Dicamba	0.5	1.03	0.66	**1.03**	2.06	**3.33**	3.2		55	110	55	110	50
MCPP	500	633	774	**774**	1549	**3333**	4.3		50	150	50	150	50
MCPA	500	162	251	**251**	502	**3333**	13.3		60	145	60	145	50
Dichloroprop	5	0.97	2.18	**2.18**	4.35	**8.33**	3.8		75	140	75	140	50
2,4-D	5	1.21	1.70	**1.70**	3.41	**8.33**	4.9		35	145	35	145	50
Silvex (2,4,5-TP)	0.5	0.07	0.054	**0.068**	0.135	**0.833**	12.3		45	125	45	125	50
2,4,5-T	0.5	0.06	0.074	**0.074**	0.147	**0.833**	11.3		45	135	45	135	50
Dinoseb	2.5	0.36	0.24	**0.363**	0.726	**8.33**	23.0		5	130	5	130	50
2,4-DB	5	1.08	0.91	**1.08**	2.16	**4.17**	3.9		50	155	50	155	50
Surrogate:													
DCAA									50	150			

Criteria for the ratio (RD) of mean amount found to MDL: >1 but <10 for soil and for MQL/MDL ratios (RQ) to be >3.

Default QC limits (50% to 150%) are used for MCPP. DoD QSM limits are available for 2,4-D, 2,4-DB, 2,4,5-T, 2,4,5-TP, Dicamba, Dinoseb, and Dichloroprop in solid matrices. The aqueous DoD QSM limits are used as interim default limits for soil matrix.

RPD limits are those described for pesticides (SW 8081) in USACE EM200-1-3, Appendix I.

Default AML QC limits are used for surrogate recoveries.

The following criteria will apply for marginal exceedances:

Number of allowed ME analytes per total analytes: 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).

NOTE: Marginal exceedance concept is not applicable to herbicides analysis. Control limits and ME limits are identical.

NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.

Table XI
List of Appendices

Appendix Number	Appendix Topic
1	Diazomethane Preparation
2	Initial Calibration (ICAL) Summary (Form-6 equivalents) Instrument Printouts
3	Calibration Verification (ICV and CCV) Summary (Form-7 equivalents) Instrument Printouts and Standard Chromatograms
4	Retention Time Window Summary
5	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
6	Method Detection Limit (MDL) Studies Summary
7	Sample Reports (Form-1 equivalents)
8	QC Association Forms (Form-4 equivalents) Sample Preparation Logsheets Instrument Run Log
9	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
10	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
11	Surrogate Recovery Reports (Form-2 equivalents)
12	Sample Results Worksheet (Form-10 equivalents)
13	DoD QSM Method Quality Objectives for Method 8151 AFCEE MQOs for Method 8151

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. O-Herbicides-RD

CHLOROPHENOXY HERBICIDES BY GC/ECD (EPA 515.1 to 515.4, and SW846-8151A)

January 17, 2006

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph. D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP), which is based on EPA methods (515.1, 515.4 and SW-846 Method 8151A) is used to determine the concentrations of various chlorophenoxy herbicides in solid and liquid matrices, using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD). This SOP is normally used for the determining the following chlorinated acid herbicides and related compounds in groundwater, wastewater, soil, sediment and waste samples. The words, method and SOP are used synonymously in this document.

Compound	**CAS Number**
2, 4-D	94-75-7
2, 4-DB	94-82-6
2, 4, 5-TP (Silvex)	93-72-1
2, 4, 5-T	93-76-5
3, 5-Dichlorobenzoic acid	51-36-5
Acifluorfen	50594-66-6
Bentazon	25057-89-0
Chloramben	133-90-4
Dacthal di-acid	
Dalapon	75-99-0
Dicamba	1918-00-9
Dichlorprop	120-36-5
Dinoseb	88-85-7
MCPA	94-74-6
MCPP	93-65-2
Pentachlorophenol	87-86-5
Picloram	1918-02-1

Because these compounds are produced and used in various forms (acid, amine, salt, ester, etc.) this SOP includes a hydrolysis step to convert the various forms to the acids form prior to derivatization such as methylation. The results therefore correspond to the total amount of herbicide compounds in the samples. Other compounds that may be analyzed by this method are 4-nitrophenol, pentachlorophenol, acifluorfen, bentazon, chloramben, DCPA diacid, 3, 5-dichlorobenzoic acid, 5-hydroxydicamba and picloram. These compounds are not analyzed on a daily basis. Therefore, method performance should be established before data can be reported for unknown samples.

1.2 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatography and in the interpretation of gas chromatograms. Only experienced analysts should be allowed to work with diazomethane due to health hazards (explosive, carcinogen) associated with this compound.

2.0 SUMMARY OF METHOD

2.1 Water (One liter) samples are acidified and extracted with ether. Soil samples (Thirty grams) are acidified, extracted with successive acetone and ether steps. Waste (One gram) is diluted with ether. The ether extracts from the various matrices is then hydrolyzed with potassium hydroxide, solvent washed with ether, acidified and re-extracted with ether. The ether extract is concentrated, derivatized with diazomethane, and solvent exchanged to hexane. Microextraction procedures can be used when detection limit requirements are met.

2.2 Analysis is by gas chromatography with dual capillary columns and electron capture detectors with internal standard calibration. The herbicides are reported as acid equivalents. The sensitivity of the method is usually determined by the level of interferences.

3.0 INTERFERENCES

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in the gas chromatograms. Method blanks are extracted with each extraction set to monitor interferences.

3.2 Glassware should be cleaned following normal cleaning procedures. Since herbicides are strong organic acids and react readily with alkaline active sites, all glassware used in the extraction procedure need further cleaning. Glassware should be rinsed with 20% HCl followed by an acetone rinse immediately before use. Glasswool should be acid rinsed as well. Anhydrous sodium sulfate used for drying soil samples should be acidified before use.

3.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from sample to sample. Organic acids, especially chlorinated acids, cause the most direct interference with this method. Phenol, including chlorophenols, chlorinated hydrocarbons, phthalate esters and oils also cause interferences. The alkaline hydrolysis and subsequent ether wash of the basic solution removes many of these interferences.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph - A Hewlett Packard 5890 Series II GC equipped with dual Ni-63 electron capture detectors is used. The system is complete with a temperature-programmable GC for splitless injection. The system includes all accessories, including syringes, analytical columns and gases. The column flow rate remains constant throughout the temperature program operation.

4.2 Gas chromatographic columns - A dual column system is employed. A 30-meter x 0.32-mm ID capillary column (primary) coated with DB-5 for a film thickness of 0.25-um (J&W Scientific) is employed. A 30-meter x 0.32-mm ID capillary column (confirmation) coated with DB-1701 for a film thickness of 0.25-um (J&W Scientific) is employed. Equivalent columns may be used in place of both columns. The primary and confirmation columns are used interchangeably.

4.3 Data system - Hewlett Packard Environquant is used for data acquisition and storage on machine-readable media of all data obtained throughout the duration of the GC program.

4.4 Microsyringes - 10, 25, 100, 250, 500 and 1000-uLs.

4.5 Balance - Analytical, 0.0001 grams and top loading, 0.1 grams.

4.6 Disposable pipettes - Pasteur

4.7 Test tubes - 5-ml to 8-ml, with Teflon lined screw caps.

4.8 Separatory funnels - 2000- mL

4.9 Kuderna-Danish (K-D) apparatus as described below:

- 4.9.1 KD Tubes
- 4.9.2 KD Evaporation flask (500-mls), attached to concentrator tubes
- 4.9.3 Three ball macro Snyder column
- 4.9.4 Clamps

4.10 Boiling Chips - solvent extracted

4.11 Heated water bath (60º to 65ºC)

4.12 Microsyringes - 10, 50, 100, 500, 1000-uLs.

4.13 Tekmar Sonic Disrupter

4.14 Glass wool - acid rinsed and dried

4.15 Stainless steel table spoons

4.16 Glassware and kit for the preparation of the diazomethane solution

4.17 Various beakers, funnels, 125-ml Erlenmeyer flasks, 24/40 ground glass 250-ml and 500-ml Erlenmeyer flasks

4.18 pH paper – 0-14 range

4.19 Mechanical shaker

4.20 Centrifuge

5.0 REAGENTS

5.1 It is assumed that all inorganic/organic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where available. Other reagents should be of high enough purity to maximize the accuracy of the determination.

5.2 Organic free reagent water - All references to water in this method should be organic free reagent water.

5.3 Solvents – Solvents used should be pesticide grade or equivalent

- 5.3.1 Diethyl ether, unpreserved and free of peroxides.
- 5.3.2 Methylene chloride
- 5.3.3 Acetone
- 5.3.4 Isooctane
- 5.3.5 Toluene

5.3.6 Carbitol –(diethylene glycol monoethyl ether)

5.3.7 Hexane

5.3.8 Methyl tert-butyl ether (MTBE) - High purity, demonstrated to be free from analytes and interferences (HPLC grade or better).

5.3.9 Hexane: MTBE (90:10, v/v) Wash Solvent – High purity, unpreserved, demonstrated to be free from analytes and interferences (HPLC grade or better).

5.4 Sodium Sulfate – granular, anhydrous. Acidify with concentrated sulfuric acid and preclean with methylene chloride. Mix 1-kg of sodium sulfate (precleaned with methylene chloride) with 300-mls acetone and 1-ml conc. sulfuric acid. Dry at 50 degrees C. Check by adding 1-g of acidified sodium sulfate to 5-mls of water. The pH should be < 4. Store in a desiccator or at 100°C to keep the reagent dry.

5.5 Copper Sulfate pentahydrate, ($CuSO_4.5H_2O$) – ACS Reagent grade.

5.6 Acid solutions

5.6.1 1:1 sulfuric acid – slowly add 50-mls conc. sulfuric acid to 50-mls of water.

5.6.2 1:3 sulfuric acid – slowly add 250-mls conc. sulfuric acid to 750-mls of water.

5.6.3 20 % HCl – slowly add 200-mls conc. HCl acid to 800-mls of water.

5.6.4 5% acidified sodium sulfate – dissolve 50-grams acidified sodium sulfate in 1-liter of water.

5.7 Potassium Hydroxide – 37% aqueous solution. Dissolve 185-grams potassium hydroxide pellets in 500-mls of water.

5.8 Diazald (n-methyl-n-nitroso-p-toluenesulfonamide)

5.9 Diazald Kit (or equivalent) – Used to prepare diazomethane solution (See Appendix 1 for diazomethane preparation).

5.10 Silicic acid – 100-mesh

5.11 Stock Standard solutions – The stock standards for instrument calibrations are purchased as certified solutions from Ultra Scientific (Catalog number HBM-8150M). All the compounds in the stock standard are in the methylated form. The concentration of each analyte (or compound) is listed in Table I. After opening the stock vial, transfer the stock solutions to a GC vial and seal with PTFE-lined screw caps. Store at 4 degrees C and away from light. Stock standards expire on the date shown by the manufacturer. Check stock standards for signs of evaporation or degradation, especially immediately prior to preparing calibration standards from them.

5.12 Intermediate Standard Solutions - From the Stock solution, prepare an intermediate solution by diluting the stock solution by a factor of 1:100. Take 1.0-ml of the stock solution and dilute it to 100-mls with hexane in a volumetric flask. The concentration of each compound in the intermediate solution, which is also used as the highest concentration calibration standard, is shown in Table

II. The intermediate standard solution should be replaced after one year or sooner if routine QC or comparisons with check standards indicate problem(s).

5.13 Calibration standard solutions – A five-point initial calibration (ICAL) curve is prepared from the intermediate standard solution. Calibration solutions should be replaced after six months or sooner if comparisons with check standards indicate a problem.

5.13.1 The intermediate solution also serves as the highest point of the curve (herbicide Std 5). Prepare four separate dilutions of the intermediate solution using the following tables to prepare a set of five calibration solutions:

5.13.2 MID HIGH POINT: Take 700-uls of the intermediate solution and dilute with 300-uls of hexane. This is herbicide Std 4 (Mid high point). The concentrations of the analytes in this solution are shown in Table III.

5.13.3 MID POINT: Take 500-uls of the intermediate solution and dilute with 500-uls of hexane. This is herbicide Std 3 (Mid point), which is described in Table IV. This solution will also serve as standard used to verify calibration (CALV) or as the continuing calibration verification standard (CCV).

5.13.4 LOW MID POINT: Take 300-uls of the intermediate solution and dilute with 700-uls of hexane. This is herbicide Std 2 (Low-Mid point), which is described in Table V.

5.13.5 LOW POINT: Take 100-uls of the intermediate solution and dilute with 900-uls of hexane. This is herbicide Std 1 (Low point), which is described in Table VI.

5.14 Surrogate solution – The compound 2, 4-Dichlorophenylacetic acid (DCAA) is used as the surrogate or system monitoring compound in this method. A shooter solution is purchased from Ultra Scientific (Catalog number PPS-164X) at a concentration of 2-ug/ml in methanol. Spike 0.5-ml of this solution into every sample including all the QC samples. Stock surrogate standards expire on the date shown by the manufacturer. Check stock standards for signs of evaporation or degradation, especially immediately prior to preparing calibration standards from them.

5.15 Matrix Spiking Solution – The MS/MSD spiking stock solution is purchased from Ultra Scientific (Catalog number HBM-8150A). The Initial concentration is the same as in the calibration stock solution (Table I) except that the compounds are in the acid form. This stock solution is diluted by a factor of 1:40 with acetone to prepare MS/MSD spiking solution. Take 1.25-mls of the stock solution and dilute to 50-mls with acetone in a volumetric flask. Spike 1.0-ml of this solution to the MS/MSD samples. When the volume of the final extract is 5-mls, the final concentrations of the analytes are shown in Table VII. Stock MS/MSD standards expire on the date shown by the manufacturer. The working MS/MSD spiking solutions should be replaced after six months or sooner if comparisons with check standards indicate a problem.

5.16 Laboratory Control Standard Spiking Solution – The LCS solution is purchased from Supelco (Catalog number 46861-U). The initial concentration of the solution is the same as the calibration stock standards except that the compounds are in the acid form. This stock solution is diluted by a factor of 1:40. Take 1.25-mls of the stock solution and dilute to 50-mls with acetone in a volumetric flask. Spike 1.0-ml of this solution to the LCS. When the final extract is at a final volume of 5-mls, the final extract concentrations will be as shown in Table VIII. Stock LCS/LCSD standards expire on the date shown by the manufacturer. The working LCS/LCSD spiking solutions should be replaced after six months or sooner if comparisons with check standards indicate a problem.

5.17 Internal Standard solution – The internal standard is 4, 4' – dibromooctafluorobiphenyl (DBOB) and is purchased from Ultra scientific (Catalog number PPS-173) at a concentration 1000-ug/ml. Spike 10-uls of this solution into each 1-ml aliquot. This gives a final concentration of 10-ug/ml in the final extracts used in instrumental analysis. Fortify calibration standards and sample extracts including dilutions with the internal standard immediately prior to analysis. Stock internal standard solutions expire on the date shown by the manufacturer.

6.0 SAMPLE PRESERVATION HANDLING & STORAGE

6.1 One liter amber glass bottles with Teflon lined screw caps may be used for collecting aqueous samples in accordance with EPA SW-846 method 8081A. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 2 bottles for each water sample to be collected. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.

6.2 One 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples in accordance with EPA SW-846 method 8081A. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each solid sample. Quality control samples such as MS and MSD should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. These samples may be shared with samples collected for other analyses such as semivolatiles (8270C), PCBs (8082), Diesel (DRO, 8015-mod.) and other pesticides (8140, etc.).

6.3 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Analytical data generated outside of the specified holding times must be considered to be minimum values only. Such data may be used to demonstrate that a waste is hazardous where it shows the concentration of a constituent to be above the regulatory threshold but cannot be used to demonstrate that a waste is not hazardous. The holding time for the extraction of aqueous samples, which includes TCLP leachates is 7 days from the date of sample collection or TCLP leachate preparation. The holding times for the extraction of soil,

sediment and waste samples, which includes TCLP leachate preparation is 14 days from the date of sample collection. The holding time for the instrumental analysis of sample extracts is 40 days from the date of extraction. Sample extracts should be stored at 4° C and protected from light.

7.0 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control contain a number of regulated compounds at high concentrations. The toxicity of these compounds present in such complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

7.5 Diazomethane is a carcinogen and can explode under certain conditions. When preparing the diazomethane solution use a hood, do not heat above 90 degrees C. Do not use ground-glass joints, sleeve bearings or glass stir rods. Store away from alkali metals. Solutions of diazomethane decompose rapidly in the presence of solid materials such as copper powder, calcium chloride and boiling chips.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Aqueous and soil samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters after the solvent(s) have been evaporated off in a hood.

9.0 PROCEDURE

9.1 Extraction of Water Samples

9.1.1 Mark the meniscus of the one liter bottle with a black magic marker. Pour the entire contents of the bottle into of separatory funnel. Add 150-mls of ether to the sample bottle and shake the bottle lightly. Pour the ether from the bottle into the separatory funnel of the sample. Fill the bottle with water to the meniscus mark. Pour this water into a graduated cylinder and record the amount in the extraction log book. Spike the samples with the appropriate surrogate solution. Remember to spike the MS/MSD and LCS with the surrogate as well as the appropriate spiking solution for the MS/MSD and LCS. Adjust the pH of the samples to less than 2 with 1:1 sulfuric acid.

9.1.2 Extract the samples by shaking the funnel for 2-minutes with periodic venting to release excess vapor pressure. Allow the organic layer to separate from the water for 10-minutes. If an emulsion forms more than one third the solvent volume, attempt to break it up with a stirring rod. Leave the emulsion in the water portion until the third extraction. After the third extraction, if more than one third is still emulsion, that extract may be frozen to separate the phases. Drain the water phase into a 2-liter Erlenmeyer flask. Collect the ether extract in a 250-ml Erlenmeyer flask. Approximately 80-mls of the ether will remain dissolved in the water phase.

9.1.3 Pour the approx. 1-liter water sample back into the separatory funnel. Extract the sample two more times using 50-mls of ether each time. Use the 50-ml of ether to rinse the Erlenmeyer flask before adding it to the 2-liter separatory funnel. Combine the ether extracts in the 250-ml Erlenmeyer flask.

9.1.4 Hydrolysis – To the 250-ml Erlenmeyer flask containing the ether extracts, add 2-mls of the 37% KOH, 15-ml reagent water and Teflon boiling chips. Attach a three-ball Snyder column. Prewet the Snyder column by adding 1-ml of ether to the top. Place in a hot water bath (60-65 degrees C). The ether will evaporate in about 15-minutes leaving the basic water. Continue to heat for a total of 60-minutes. Remove the extracts and let them stand at room temperature for 10-minutes before removing the Snyder columns.

9.1.5 Solvent Cleanup – Quantitatively transfer the basic water solution to a 125-ml separatory funnel. Check the pH. It should be >12. Add more 37% KOH if the pH is below 12. Wash the basic solution twice by shaking it for 1-minute with 20-ml portions of ether. Drain the water solution back into the 250-ml Erlenmeyer. Discard the ether phase. Pour back the water solution using the 20-ml of ether to rinse the 125-ml funnel. The herbicides will remain in the basic water solution as potassium salts.

9.1.6 Re-extraction with ether – Quantitatively transfer the basic water solution back to the 125-ml sep. funnel. Acidify the solution to a pH 2 by adding 2-ml of cold 1:3 sulfuric acid. Check the pH. Add 20-ml of ether and extract the solution by vigorously shaking the funnel for 2-minutes. Drain the water layer into a 125-ml flask. Drain the ether layer into a 250-ml 24/40 ground glass neck flask. Extract the water solution two more times with 10-ml portions of ether. Use the 10-ml portion of ether to rinse the 125-ml funnel combining the extracts in the 250-ml 24/40 flask. Add sufficient acidified sodium sulfate to each extract so that some remains free-flowing. Stopper and allow the extracts to remain in contact with the sodium sulfate at least 4-hours or refrigerate overnight.

9.1.7 Concentrate – Concentrate the extract by a Kuderna-Danish apparatus and N-Evap. Transfer the ether extract into the receiver through a funnel plugged with acid washed glass wool and topped with acidified sodium sulfate. Use liberal washings with ether to complete the transfer. Use a

glass rod to crush any caked sodium sulfate and rinse with ether into the receiver. Concentrate the extract to approx. 3-ml.

9.1.8 Methylation – The methyl esters can be formed with diazomethane by adding a diazomethane solution. Add 0.1-ml of MeOH to each extract and dilute with 1-ml of ether. Slowly add 2-mls of diazomethane solution to each extract. Let the extract stand for 10-minutes with occasional swirling. The yellow color must persist during this 10-minute process.

9.1.9 Solvent exchange – N-Evap the extract to 1-ml using a gentle stream of nitrogen. Add 1-ml of hexane and continue to nitrogen blow down. Do not let the extract go dry. Continue this process three more times. The yellow color will disappear during the blow-down. Add 0.1-grams of silicic acid to the extract. Dilute the extract to a final volume of 5-ml with hexane.

9.2 Micreoextraction of Water Sample

When microextraction is employed, initial, continuing and second source calibration standards are processed using the procedures described below for environmental samples. Calibration standards are prepared using procedures described for the preparation of spiked samples with the exception that smaller quantities (0.1 L) are prepared and a minimum of five concentration levels are employed for the initial calibration.

9.2.1 Remove the samples from storage and allow them to equilibrate to room temperature. Place 40 mL of the water sample into a precleaned 60-mL, glass vial with a PTFE lined screw cap using a graduated cylinder.

9.2.2 Add 10 uL of surrogate standard (100 ug/mL, 2, 4-dichlorophenylacetic acid in acetone to the aqueous sample.

9.2.3 Add 1 mL, of the 4 N NaOH solution to each glass vial. Check the pH of the sample with pH paper or a pH meter. If the sample does not have a pH greater than or equal to 12, adjust the pH by adding more 4 N NaOH solution. Let the sample sit at room temperature for 1 hour, shaking the contents periodically NOTE: Since many of the herbicides contained in this method are applied as a variety of esters and salts, it is vital to hydrolyze them to the parent acid prior to extraction. This step must be included in the analysis of all extracted field samples, LCS and MS, and calibration standards associated with the microextraction procedure.

9.2.4 Following hydrolysis, add 5 mL of (90:10, v:v) hexane:MtBE and shake vigorously for three minutes. Allow the phases to separate for approximately 5 minutes then remove and discard the top hexane/MtBE layer. This wash aids in sample cleanup and removes any Dacthal from the sample which would interfere with the quantitation of the Dacthal metabolites.

9.2.5 Adjust the pH to approximately 1 by adding concentrated sulfuric acid. Cap, shake and then check the pH with a pH meter or narrow range pH paper. Add additional sulfuric acid as needed to properly adjust the pH.

9.2.6 Quickly add approximately 2 g of copper II sulfate pentahydrate and shake until dissolved. This colors the aqueous phase blue and allows the analyst to better distinguish between the aqueous phase and the organic phase in this micro extraction.

9.2.7 Quickly add approximately 16 g of muffled sodium sulfate and shake until almost all is dissolved. Sodium sulfate is added to increase the ionic strength of the aqueous phase and thus further drive the chlorophenoxyacids into the organic phase. The addition of salt also decreases the solubility of MtBE in the aqueous phase and allows greater volumetric recovery. The addition of this salt and the copper II sulfate pentahydrate should be done quickly so that the heat generated from the addition of the acid will help dissolve the salts.

9.2.8 Add exactly 4.0-mL MtBE and shake vigorously for three minutes. Allow the phases to separate for approximately 5 minutes. Using a Pasteur pipette, transfer the sample extract (upper MtBE layer) to a 7- mL, screw cap vial. Add 0.6 g acidified sodium sulfate and shake. This step is included to dry the MtBE extract.

9.2.9 Using a Pasteur pipette, transfer the extract to a second, 7-mL glass vial. Add 250 uL of the diazomethane solution to each vial. The contents of the vial should remain slightly yellow in color indicating an excess of diazomethane. Additional diazomethane may be added if necessary. Let the esterification reaction proceed for 30 minutes. Remove any unreacted diazomethane by adding 0.1 g of silica gel. Effervescence (evolution of nitrogen) is an indication that excess diazomethane was present. Allow the extracts to sit for 0.5 hour.

9.2.10 Transfer the extract to an autosampler vial. A duplicate vial may be filled using the excess extract. Analyze the sample extracts as soon as possible. The sample extract may be stored up to 21 days if kept at 0 °C or less. Keep the extracts away from light in amber glass vials with PTFE lined caps.

9.3 Extraction of soil, sediment or other solid samples.

9.3.1 Weigh approx. 30-grams of wet sample into a 500-ml wide mouth Erlenmeyer flask with a 32 ground glass neck. Add all spiking solutions accordingly. Acidify the samples with conc. HCL (2-3 mls) to a pH of 2. Allow the samples to stand for 15-minutes occasionally stirring until the pH remains below 2. Add more acid if necessary.

9.3.2 Add 20-ml of acetone to each sample, stopper and place on a shaker for 20-minutes. Add 80-mls of ether to each sample and shake for another 20-minutes.

9.3.3 Remove the flasks from the shaker and allow the solids to settle. Decant the solvent layer into a 100-ml flask. If the extraction does not have a decanted volume of at least 75-ml, an additional extraction is necessary. Pour the extracts into a 2-liter separatory funnel.

9.3.4 Check the pH of the sample to ensure that the pH remains at 2. If the pH is not at 2, add more HCl until it is stabilized. Perform two more extractions of the sample as in 9.2.2 and collect the extracts in the flasks.

9.3.5 Add the extracts and 250-mls of 5% acidified sodium sulfate solution to 2-liter separatory funnel. Gently mix the contents of the funnels for 1-minute and allow the phases to separate. Drain the water/acetone phase

into 500-ml flasks and the ether phase into a separate 500-ml flask with 24/40 ground glass necks. Pour back the water/acetone phase and re-extract with 25-mls of ether. Combine the ether extracts.

9.3.6 Hydrolysis - To the 500-ml Erlenmeyer flask containing the ether extracts, add 5-mls of the 37% KOH, 30 ml reagent water and Teflon boiling chips. Attach a three-ball Snyder column. Prewet the Snyder column by adding 1-ml of ether to the top. Place in a hot water bath (60-65 degrees C). The ether will evaporate in about 20-30 minutes leaving the basic water. Continue to heat for a total of 90-minutes. Remove the extracts and let them stand at room temperature for 10-minutes before removing the Snyder columns.

9.3.7 Solvent Cleanup - Quantitatively transfer the basic water solution to a 125-ml separatory funnel. Check the pH. It should be >12. Add more 37% KOH if the pH is below 12. Wash the basic solution twice by shaking it for 1-minute with 40-mls portions of ether. Drain the water solution back into the 500-ml Erlenmeyer. Discard the ether phase. Pour back the water solution using the 20-ml of ether to rinse the 125-ml funnel. The herbicides will remain in the basic water solution as potassium salts.

9.3.8 Re-extract with ether - Quantitatively transfer the basic water solution back to the 125-ml Sep funnel. Acidify the solution to a pH 2 by adding 5 ml of cold 1:3 sulfuric acid. Check the pH. Add 40-ml of ether and extract the solution by vigorously shaking the funnel for 2-minutes. Drain the water layer into a 125-ml flask. Drain the ether layer into a 250-ml 24/40 ground glass neck flask. Extract the water solution two more times with 20-ml portions of ether. Use the 20-ml portion of ether to rinse the 125 ml funnel combining the extracts in the 250-ml 24/40 flask. Add sufficient acidified sodium sulfate to each extract so that some remains free-flowing. Stopper and allow the extracts to remain in contact with the sodium sulfate at least 4-hours or refrigerate overnight.

9.3.9 Concentration - Concentrate the extract by a Kuderna-Danish apparatus and N-Evap. Transfer the ether extract into the receiver through a funnel plugged with acid washed glass wool and topped with acidified sodium sulfate. Use liberal washings with ether to complete the transfer. Use a glass rod to crush any caked sodium sulfate and rinse with ether into the receiver. Concentrate the extract to approx. 3-ml.

9.3.10 Methylation – The methyl ester scan be formed with diazomethane by adding a diazomethane solution. Add 0.1-ml of MeOH to each extract and dilute with 1-ml of ether. Slowly add 2-mls of diazomethane solution to each extract. Let the extract stand for 10-minutes with occasional swirling. The yellow color must persist during this 10-minute process.

9.3.11 Solvent exchange – N-Evap the extract to 1-ml using a gentle stream of nitrogen. Add 1-ml of hexane and continue to nitrogen blow down. Do not let the extract go dry. Continue this process three more times. The yellow color will disappear during the blow-down. Add 0.1-grams of

silicic acid to the extract. Dilute the extract to a final volume of 5-ml with hexane.

9.4 Extraction of Waste samples.

9.4.1 If the waste sample is miscible with ether, weigh 1 to 2-grams into a 250-ml flask with 24/40 ground glass necks. Add all spiking solutions accordingly.

9.4.2 If the waste sample is not miscible with ether, try various solvents that are also miscible with ether to dissolve the sample. After the correct solvent combination is found, weigh 1 to 2-grams into a 250-ml flask with 24/40 ground glass necks. Add all spiking solutions accordingly.

9.4.3 Add 100-mls of ether to the flask.

9.4.4 Proceed with sections 9.2.6, 9.2.7, 9.2.8, 9.2.9, 9.2.10 and 9.2.11.

9.5 Instrument Calibration and Analysis

9.5.1 At the initiation of this method and thereafter, whenever continuing calibration fail criteria or when equipment changes are made (such as a new detector or new column is installed), establish a 5 point initial calibration (ICAL) standard curve. Additional ICAL standards may be analyzed for additional options for generating valid ICALs for each compound.

9.5.2 Prepare a 5-point curve (ICAL) by injecting the calibration standards described in Section 5.12. Spike 10-uls of the internal standard (DBOB) into each 1- ml calibration solution prior to injection into the GC/ECD. Calculate response factors (RF, see equations below) from the areas for the internal standard and the analyte peaks for each compound at each concentration level. Calculate the average response factor (RF_m) and the associated standard deviation (SD) from the five RFs obtained for five concentration levels.

9.5.3 Linearity – If the % RSD of any compound is 20% or less, then the relative response factor is assumed to be constant over the calibration range and the average relative response factor may be used for quantitation.

9.5.4 If the % RSD of any detected compound is greater then 20%, use least square regression methods to calculate sample concentrations. Non-linear regression procedures may require more than 5 points (minimum for ICAL). For regression methods, the instrument response is treated as the dependent variable (y) and the standard amount is the independent variable (x). When "Cs" is used as "x", use "As*Cis/Ais" as "y". When "Cs/Cis" is used as "x", use "As/Ais" as "y". Do not include origin (0,0) as an extra calibration point.

9.5.5 Weighted least squares method is preferred for calculating concentrations more accurately at lower levels. A linear least squares equation (y = a*x + b) is preferred over non-linear calibration equations (y = a*x2 + b*x + c). The coefficient of determination (COD), coefficient of variation (r2) or

correlation coefficient (r) should be used to measure the goodness of fit. For regression models to be used for quantitative purposes, the COD, r2 or r must be greater than or equal to 0.99. A value of 1.00 indicates a perfect fit. Corrective action may be required if the criteria for %RSD, r, r2, or COD are not met.

9.5.6 Document ICAL in Form-6 equivalents, or in linear regression charts (See Appendix 2 for details). Use the following calculations for determining response factors and determining %RSD:

$RF = (A_s C_{is}) / (A_{is} C_s)$

where:
A_s = Area of the compound being measured
A_{is} = Area of the specific internal standard
C_{is} = Concentration of the specific internal standard
C_s = Concentration of the compound being measured

$\% RSD = ((SD)/(RF_m))*100\%$

where:

RF_m = mean of the 5 initial RFs for a compound
SD = standard deviation of the 5 initial 5 RFs for a compound.

Where SD is the square root of the following equation:

$$(SD^{**}2) = \frac{\sum_{i=1}^{n} (RF_i - RF_m)}{(n-1)}$$

n = number of levels of standards

9.5.7 An initial calibration (ICAL) should be considered a single event process. Consult the QA office or check applicable EPA methods (e.g. SW-846 Method 8000C and 8081A) for additional guidance on appropriate corrective action.

9.5.8 Re-fitting of ICAL – The selected model for ICAL may be subjected to an additional re-fitting check to establish representativeness of the model for each compound in each ICAL standard. This is an optional procedure, which is recommended but not required since automated data processing procedures may not be able to accommodate it. Calculate the %D between the calculated amount (Cc) for each compound in each ICAL standard from the ICAL model and its expected value (Ce). Ideally, the %D ((Cc-Ce) * 100 /Ce) for each compound in ICAL standard should meet the QC criteria for %RSD. It is possible for one or more ICAL

standards to have %D exceeding the %RSD criteria, while the %RSD for ICAL model is within QC limits. The re-fitting procedure may be used to identify ICAL standards that need re-analysis or dropped from the calculation of ICAL model when re-analysis was not performed.

9.5.9 Check and optimize instrument operating conditions including automated peak integration procedures for appropriateness in addressing the chromatographic needs of the specific compound with problems.

9.5.10 If the problem appears to be associated with just one of the ICAL standards, that one standard may be reanalyzed once immediately (must be within 8-hours) after the ICAL. Discard all the results from the original analysis of the standard in question and replace it with the results from re-analysis.

9.5.11 When additional calibration standards (six or seven instead of five) are analyzed, narrowing the range of concentrations to be used for determining the goodness of fit is an acceptable procedure. Five or more standards should remain for the calculation of RSD or linear regression for each compound after one or more points are removed from the ICAL. More points (6 for second order and 7 for third order) may be needed for non-linear regression. It is generally prohibited to remove data points from within a calibration range, while still retaining the extreme ends of the calibration range. Do not discard a middle point unless it appears to have an obvious problem and six or more standards were analyzed for ICAL and re-analysis of the affected standard was not performed within the required time limits. Changes to the upper end of the calibration range will affect the need to dilute samples above the range. A change to the lower range is not recommended since it will affect the method quantitation limit (MQL) used to report data. The default MQLs, which are based on the lowest ICAL concentration are stored in the LIMS and project-specific changes are not practical.

9.5.12 The refitting procedures may identify failed %Ds for upper level calibration standards for average response factor methods. The analyst may take alternate corrective actions such as the analysis of sample extracts under dilution for the affected compounds.

9.5.13 The refitting procedures may lead to failed %Ds for lower level calibration standards for regression methods. The analyst may take appropriate corrective action such as flagging the sample result as an estimated amount ("J" flag) at the lower level encountered in the sample.

9.5.14 Grand mean RSD – Previous versions of the EPA method(s) had provisions for the calculation of the grand mean %RSD for all target compounds in the ICAL solution, when the % RSD of any detected compound is greater than 20%. The grand mean %RSD should be <20% for ICAL to be acceptable. This procedure is discontinued. Criteria for Initial calibration are compound-specific.

9.5.15 The term "calibration verification" (CALV) is used to describe initial verification (ICV) of the initial calibration (ICAL) using second-source standards and subsequent continuing verifications (CCVs) using same-

source standards. The response factors calculated during CALV are not used for sample quantitation since CALV standards are not used as "continuing calibration" standards permitted in some of the EPA methods.

9.5.16 Initial Calibration Verification (ICV) - Analysis of a second source ICV standard is required before any samples can be analyzed for USACE projects. Since second source standards are not employed for the analysis of LCS/LCSD and MS/MSD samples, ICV analysis is applicable for all projects. Calculate the percent difference (%D) or %Drift for the observed response factor in ICV from the mean RF as described for CCV. The %D or %drift should not exceed 15% for each target compound. Document calibration verification in instrument printouts as shown for CCVs. Check the ICAL and ICV standards if it is not achievable for all the target compounds without any exception. Use professional judgment for non-USACE projects and for compounds that are not contaminants of concern (COC) for USACE projects. The ICV may be used as the beginning CCV for sample analysis.

9.5.17 After the initial calibration curve has been established and verified with an ICV, analytical sample may be analyzed. A continuing calibration check (CCV) can be made, by injecting the mid-point calibration solution (as described in Section 5.12.3) after every 10 injections. The percent difference (%D) for the RF from the continuing calibration check must be within 15% of the mean RF (RF_m) obtained during the initial calibration (ICAL). Document calibration verification in Form-7 equivalents (See Appendix 3 for details). The % difference is calculated as follows:

$$\% D = ((RF_v - RF_m) * 100) / RF_m$$

where:

RF_m = Average RF for the compound from ICAL
RF_v = Measured RF for the compound in CALV (ICV or CCV).

9.5.18 If the %D for any compound is greater than 15%, the GC should be restored to proper operating condition before re-analyzing the CCV for all the compounds. A new ICAL should be generated if the CCV does not meet QC criteria.

9.5.19 Grand mean %D – Previous versions of the EPA method(s) had provisions for the calculation of the grand mean %D for all target compounds in the CCV standard, when the %D of any detected compound is greater than 15%. The grand mean %D should be <15% for CCV to be acceptable. This procedure is discontinued. Criteria for CCV are compound-specific.

9.5.20 Calibration Factors are used when external standards are used for calibration. This procedure is not used at AML and they are described only when they are necessary. Situations include internal standard coeluting with one of the target compounds in one or more columns. When external standard calibration is employed, calculate the calibration

factor for each analyte at each concentration, the mean calibration factor, and the relative standard deviation (RSD) of the calibration factors. If external standard calibration is employed, refer to Method 8000C or AML SOP on organochlorine pesticides or PCBs (8081 0r 8082) for the calculation of calibration factors.

9.5.21 Retention time windows are established by averaging the retention times, preferably over a 3-day period. Center the window around the daily calibration standard. Continuing calibration standards retention times may shift not more than twice the recovery window for the run to be considered valid. Any shift in retention times must be considered when evaluating sample results. Determine and document retention time window for each compound in logbooks (See Appendix 4 for details). Wider windows may be used to minimize false negatives. However, the window should not be too wide since false positives may result.

10.0 DATA ANALYSIS AND CALCULATIONS

10.1 The internal standard technique is used to determine the concentration of an identified target compound. For water samples the calculation is as follows:

$$\text{Concentration } (\mu g/L) = \frac{(As) * (IS) * (Ve)}{(Ais)\ (RFm)\ (Vs)}$$

where:

As = Area for compound being measured.
IS = Concentration of the internal standard in the extract injected (µg/mL)
Ais = Area for the internal standard
RFm = Mean Response factor for compound being measured
Vs = Volume of water (L) extracted
Ve = Volume of the final extract (mL), taking into account any dilutions made

10.2 For soil samples the calculation is as follows:

$$\text{Concentration } (\mu g/kg) = \frac{(As) * (IS) * (Ve) * (100)}{(Ais) * (RFm) * (Ws) * (\%S)}$$

where:

As = Area for compound being measured.
IS = Concentration of the internal standard in the extract injected (µg/mL)
Ais = Area for the internal standard
RFm = Mean Response factor for compound being measured
Ws = Wet weight of soil sample (kg) extracted

Ve = Volume of the final extract (mL), taking into account any dilutions made

10.3 Calculate the matrix spike (MS) and/or MS duplicate (MSD) recoveries for each analyte from the following formula:

$$\text{Recovery} = \%R = \frac{Cs - Cu}{Ct} * 100$$

Where
Cs = Measured concentration of the analytes in the spiked sample
Cu = Measured concentration of the analytes in the unspiked sample
Ct = True value or spike concentration of the analytes

10.4 Calculate the laboratory control sample (LCS) and/or LCS duplicate (LCSD) recoveries for each analyte using the following equation:

$$\text{Recovery} = \%R = \frac{Cs}{Ct} * 100$$

Where
Cs = Measured concentration of the analytes in the LCS/LCSD
Ct = True value (spike amount) concentration of the analytes in the LCS/LCSD

10.5 Precision can be estimated from the relative percent difference (RPD) of the two sample results obtained for the spiked samples (MS & MSD and LCS and LCSD). Calculate the relative percent difference from the following formula:

$$RPD = \frac{|R1 - R2|}{\{R1+R2\}/2} * 100$$

where
R1 = Measured sample result of the first sample aliquot
R2 = Measured sample result of the second sample aliquot

10.6 Surrogate – Spike every sample and QC sample in an analytical QC batch. Calculate surrogate recoveries using the following formula:

$$\%\text{Recovery} = \frac{\text{conc. of surrogate in the sample}}{\text{true value for the conc. of the surrogate}} * 100$$

11.0 QUALITY CONTROL

11.1 Sample preparation logs, sample data and quality control data should be gathered prior to review. Checklists for documenting raw data and their verification (See Appendix 5 for details) should be used to document verification of calculations and adherence to method procedures. Calculated sample results for each compound should be compared to their method detection limits (MDL), lowest level for reporting (LLR), reporting limits (RL) and method quantitation limits (MQL) as shown in Tables IX and X. Exceptions should be documented. Samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on these checklists. Summarize sample results using Form-1 equivalents (See Appendix 7 for details). Summarize QC results in Form-1 equivalents and recoveries and RPDs using Form-3 equivalents for MS/MSD (See Appendix 9 for details) and LCS/LCSD (See Appendix 10 for details). Use data verification and validation procedures described in detail in Section 12 for appropriate corrective action that may be required if QC elements are not in control.

11.2 Surrogate recovery should be within QC limits for samples, spikes and blanks. Document recoveries using Form-2 equivalents (See Appendix 11 for details). The laboratory currently uses QC limits from the Department of Defense Quality Systems Manual (DoD QSM, Final Version 2, June 2002) for surrogate recoveries. Flag recoveries that are outside the QC limits with an asterisk (*). If re-extraction and/or reanalysis have been performed, and the surrogate recoveries for reanalysis are within QC limits, report data based on acceptable surrogate recoveries. If the surrogate recoveries are outside the QC limits for the reanalysis, report data for both analyses with surrogate recovery flags.

11.3 Method blanks (MB) should be free of target compounds. Report target compounds that are present in the MB at levels greater than the reporting limit (RL) using J qualifiers if the levels are less than method quantitation limits (MQL). The action level for method blank is one half of the MQL. See tables IX and X for a list of MDLs, RLs, and MQLs. Document method blank or QC association using Form-4 equivalents (See Appendix 8 for details). Samples associated with the method blank containing target compounds at levels greater than on half the MQL will require re-extraction and reanalysis unless other criteria acceptable to the client are used. Since method blanks may be contaminated when they are associated with samples containing high levels of target compounds, only samples that contain less than one tenth of the MB amount may need to be re-extracted and reanalyzed.

11.4 LCS recoveries should be within QC limits (DoD QSM) for the target compounds. Flag recoveries that are outside the control limits with “ME” and compounds outside the ME limits with “ME*”. See Tables IX and X for LCS recoveries and QC limits. Contact supervisor for appropriate corrective action if more than the allowed numbers of compounds are outside the control limits or the marginal exccedance (ME) limits. Data qualification, re-extraction, and/or reanalysis of associated samples may be necessary depending on project requirements, turnaround time and other constraints.

11.5 MS recoveries should be within QC limits (DoD QSM) for the target compounds. Flag recoveries that are outside the control limits with “ME” and compounds outside the ME

limits with “ME*”. See Tables IX and X for spike recoveries and QC limits. Contact supervisor for appropriate corrective action if more than 10% of the compounds are outside the QC limits. Data qualification, re-extraction, and/or reanalysis of associated samples may be necessary depending on project requirements, turnaround time and other constraints.

11.6 Report target compounds that are present at levels greater than the method detection limit (MDL), lowest level for reporting (LLR) or the reporting limit (RL) as appropriate (See Project Plan for details) using "J" qualifiers if the levels are less than MQL. Compounds that were detected in the associated MB should also be qualified with a "B" code. If the concentration of any of the target compounds exceed the upper limit of calibration (ICAL), flag the affected compounds data with an estimated or "E" flag. The extracts may need to be diluted and reanalyzed until the concentrations are within the calibration limits for normal turnaround analyses. For rapid turnaround analyses sample results may be reported with "E" qualifiers if sufficient time is not available for reanalysis. Prepare Form-1 equivalents (See Appendix 7) for sample reports.

12 **DATA VERIFICATION AND VALIDATION -** This section will provide guidance and procedures to assess if data produced using this SOP are valid and legally defensible. Document review and comments on the checklist (See Appendix 5). Verify that sample preparation logs are complete and the instrument run logs are available for review (See Appendix 8). Anomalies and exceptions should be documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on the checklists. Army (USACE) and Air Force (AFCEE) projects may have special requirements (See Appendix 13) that will require extra attention. A simple, universal procedure for evaluating and choosing appropriate corrective action doe not exist to deal with situations when one or more QC parameters are outside the acceptance window. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints.

12.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) projects may have additional requirements that are related to USACE laboratory validation (CX), USACE District requirements, government contract requirements, specific USACE project requirements or USACE Architect/Engineer (A/E) requirements. USACE-CX requirements (Summary of Method Quality Objectives for Method 8151), AFCEE requirements and the DoD QSM requirements are included in Appendix 13.

12.1.1 The USACE requirements shown in Appendix 13 and elsewhere in this SOP are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may or may not be attainable for routine sample analysis. Since USACE MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost

constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

12.1.2 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality objectives and/or other QC requirements. Exceptions and data qualifications necessary should be documented on the checklists and eventually on case narratives.

12.1.3 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist, perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete. For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete. For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director will review the checklists (initial/date as necessary), prepare case narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

12.1.4 The blank criteria shown in Appendix 13 has been modified by USACE auditor(s) as one half of the applicable MQL (personal communications, USACE auditor, 1999).

12.2 HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

12.2.1 WATER - The holding times for sample extraction is 7 days from sampling or from the date of TCLP leachate preparation.

12.2.2 SOIL – A 14 day holding is used for soil and waste samples.

12.2.3 TCLP – TCLP leachates should be prepared within 14-days of waste sample collection. TCLP leachates should be extracted within 7 days after TCLP leachate preparation.

12.2.4 Sample extracts should be analyzed within 40-days from the date of sample extraction. This applies to extracts from aqueous, soil and leachate samples.

12.2.5 Extraction holding times are established by comparing the sampling dates on the chain of custody with the date of sample preparation. TCLP holding times are established by comparing first the COC to the leaching date and then the leaching date to the sample preparation date. Analysis holding times are calculated by comparing the extract injection dates for the instrumental analysis with the sample extraction dates on the sample preparation log.

12.2.6 ACTION – If holding times have been exceeded, contact the client. All positive results may require qualification as estimated (J) by the user (client). Include a

statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or invalid (I).

12.3 INITIAL CALIBRATION – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the GC/ECD is capable of producing data within a range that is linear. The ICAL should be verified (ICV) using a second-source standard for USACE and AFCEE projects (See Appendix 13 for details).

12.3.1 Compounds – Check to see if the mean RF for any compounds is outside the criteria of 20% or less for %RSD. If the %RSD for a compound is outside the 20% criteria, check to see if the mean RSD for all the target compounds is less than 20%. If mean RSD is less than 20%, check to see if linear regression can be used for compounds with %RSD greater than 20%. The limit for the correlation coefficient (r) used in linear regression is 0.990.

12.3.2 ACTION – Positive results for compounds not meeting the criteria may need to be qualified as estimated (J) by the user (client). A new calibration may need to be constructed if a significant number of compounds need qualification. Check the standard solutions and the instrument for problems, correct any identified problem(s) and repeat initial calibration, if necessary. For compounds with QC problems, report detects and non-detects without qualifiers and mention the outliers in the case narrative.

12.4 INITIAL CALIBRATION (ICAL)– Instrument calibration (See Appendix 3 for details) criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the GC/ECD is capable of producing data within a range that is linear. The ICAL includes initial verification (ICV) of the ICAL with a second source standard for USACE and AFCEE projects (See Appendix 13 for details).

12.4.1 CHECK – Check to see if any other compounds are outside the criteria of 20% or less for %RSD. If a compound is outside the 20% criteria, check to see if regression methods are used. The goodness of fit should be measured with the coefficient of determination (COD) or related quantities (r2 and r) greater than 0.99.

12.4.2 ACTION – Check and correct the standards, including the range of concentrations for appropriateness and repeat the ICAL analysis.

12.4.3 ACTION - Report the problem to the Project Manager and report detects and non-detects with appropriate qualifiers for rapid TAT projects. Mention the ICAL anomaly in the case narrative.

12.5INITIAL CALIBRATION VERIFICATION – Calibration verification includes initial calibration verification (ICV) and continuing calibration verifications (CCVs). A second source ICV is used to check the integrity of the ICAL standards used for calibrating the instrument. The 12-hour analytical shift begins with the injection of the ICV (instead of a CCV) for sample analyses that are performed immediately after an ICAL.

12.5.1 Target Compounds - Check to see if %D for any of the target compounds is outside the USACE QC criteria (<±15%).

12.5.2 ACTION - Check the ICAL and ICV standards for integrity. Replace one or more of the working standards (ICV first and ICAL later) for trouble shooting. Correct the problem(s) and repeat ICV analysis. Use professional judgment for non-USACE projects.

12.5.3 ACTION - When ICAL and ICV are acceptable based on SW-846 criteria, positive results and non-detects for target compounds not meeting the ±15% %D may be reported without data qualifications for non-USACE projects provided a valid CCV was obtained immediately following the CCV. The ICAL and/or ICV standards should be inspected and replaced, if necessary for compliance with this requirement for USACE projects.

12.6 CONTINUING CALIBRATION VERIFICATION – The continuing calibration verification (See Appendix 4 for details) is used to check that the instrument is still operating under the linear range of the initial calibration and has remained stable every 12 hours.

12.6.1 IS area counts (use mean IS area from ICAL for the %D of IS area) must not vary by more than a factor of 2 (-50% or + 100%) from the associated ICAL. Check to see if the %D for any compounds is outside the criteria of 15% or less.

12.6.2 %D for Compounds present in the sample – The %D for the CCVs before and after the sample analysis should be within QC limits when internal standards are not used. The %D for the CCV before sample analysis should be within QC limits when internal standards are used.

12.6.3 The %D for Compounds not present in the sample – The %D for the CCVs before and after the sample analysis may be >(+15%). Corrective action is not required unless the %D is < (-15%).

12.6.4 ACTION – Corrective action must be performed for positive results with %D outliers. In exceptional cases (e.g. rapid TAT analysis for screening level data), positive results for compounds not meeting the criteria may be qualified as estimated (J) by the user (client).

12.6.5 ACTION - Report non-detects without qualifiers and include a description of the anomaly in the case narrative.

12.6.6 ACTION – Perform more frequent CCVs (e.g. every 10 samples) than required (every 12-hours) to avoid extensive re-analysis.

12.7 METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Sample data qualification to alert blank contamination is performed by the laboratory. Sample data qualification based on all the blank results (laboratory MB, equipment blank, and other field blanks) is performed by the user (client). The processing of blanks helps eliminate reporting of false positives in project reports.

12.7.1 Method blanks should be free of all target analytes and other interfering non-target analytes. To be acceptable, blank results should not exceed one half of the applicable MQL for any compound.

12.7.2 Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples. If levels greater than one half the MQLs are present, inspect and correct the problem. Reanalyze all associated samples. Contact client if other criteria are applicable. The only exception may be quick turn around projects.

12.7.3 ACTION – All positive results are reported with a "B" code, if contaminants are found in the method blank.

12.7.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, then no action is taken.

12.7.5 ACTION – Any compound detected in the sample which were also detected in associated method blank at levels greater than one half the MQLs should be re-extracted unless other client criteria are applicable.

12.7.6 ACTION – If contaminants resulting in GC/ECD peak saturation are present, all affected compounds are qualified as invalid (I).

12.8 SURROGATE RECOVERIES – Surrogates or system monitoring compounds are different structurally, but similar chemically to classes of target compounds. These surrogate compounds are added just prior to extraction of the samples and the percent recoveries of the surrogates are calculated and evaluated.

12.8.1 Verify that the surrogate recoveries (See Table IX and X) for the surrogate compound is within the DoD QSM control limits.

12.8.2 ACTION – When surrogate recoveries are outside control limits the sample must be re-analyzed. Reanalysis with or without cleanup may be performed under dilution, if necessary if original analysis should indicate high levels of target and non-target analytes.

12.8.3 ACTION - Reextraction and cleanup will be necessary if reanalysis does not resolve the problem. If surrogate recoveries are still outside the limits, report both sets of data and describe the problem in the case narrative.

12.9 LABORATORY CONTROL STANDARD (LCS) – The analysis of LCS samples is used to evaluate method competence in the absence of matrix effects. The LCS is evaluated by comparing the percent recovery for all of the target analytes to the laboratory QC limits, which are identical to the 3-sigma DoD QSM QC limits. See Appendix 10 for guidance on documenting LCS and LCSD concentrations and %R in QC summary report forms. The DoD QSM specifies the number of compounds that can exceed the 3-sigma QC limits based on statistical considerations. The LCS recovery for such compounds should not exceed the marginal exceedance 4-sigma (ME) limits. Evaluation procedures may include comparison of LCS recoveries with those determined by project-specific DQOs (See project QAPP, if available), laboratory statistical control limits, or USEPA guidance. Initially, the effect of QC failure on the samples should be evaluated. Regardless of this assessment, steps shall be taken to find the source of the problem and correct it.

12.9.1 CHECK - LCS and LCSD recoveries (See Appendix 8 for details) should be within QC limits (See Tables IX and X). Compare the %R results for each compound in LCS with QC limits (control and marginal exceedance) to see if all

the results are within the acceptable range. Flag outliers with "ME" if it is within the 3-sigma limits. Flag it with "ME*" if it is outside the 4-sigma ME limits.

12.9.2 CHECK - Compare observed %R results for each compound with the laboratory limits for LCS/LCSD recoveries (e.g. USACE criteria). Perform manual calculation for %R, if necessary for QC outliers. Document the number of compounds that exceeded the QC limit and compare it against the maximum recommended by DoD QSM. Document this review in checklists and in case narratives.

12.9.3 ACTION - Typically, the LCS would be reanalyzed for the failed analytes (ME*) only. If the second analysis fails, then the LCS, method blank, and all associated samples of the batch would be reanalyzed for the failed analytes only. If sufficient sample is not available for reanalysis, or if the corrective action is ineffective, discuss the situation with the client project manager (or chemist). Include a discussion of the situation in the case narrative. The case narrative should discuss the corrective action taken and any other information.

12.9.4 ACTION - For non-USACE projects, no action may need to be taken on LCS and/or LCSD recovery data alone either to reanalyze samples and/or to qualify associated samples. Providing data of known quality is sufficient for most projects. If significant deviations (e.g. number of target compound exceeding QC limits for recoveries is greater than the allowable maximum) are noticed, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements, which may be compound specific. Check with the project manager about such requirements. Professional judgment may be used by the user (client) in evaluating sample data qualification options in conjunction with other QC data for the project.

12.10 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike recovery data are used to evaluate the accuracy of the analytical method on project samples. The evaluation is performed by comparing the %R for target analytes to the DoD QSM limits established for the LCS samples.

12.10.1 Document MS/MSD recoveries in instrument printouts and in LIMS generated reports as shown in Appendix IX. Flag QC outliers with "ME" and "ME*" on client reports. The MS/MSD percent recoveries should be within QC limits (Tables IX and X). Perform one manual calculation using raw data and extraction sheet information.

12.10.2 MS data evaluation is more complex than method blank or LCS data evaluation since they are complicated by matrix effects in addition to sample preparation and analysis errors. The heterogeneity of grab soil samples, and potential non-representativeness of sequentially collected water samples, etc. further complicate the evaluation since it is assumed that the native concentrations are constant in triplicate analyses. In addition, concentrations of the target analytes in the sample can also far exceed spike amounts. If the native concentration of target analytes in the sample chosen for spiking is high relative to the spiking concentration, the differences in the native concentration between the unspiked sample and the spiked samples may also become very significant.

12.10.3 In addition to their reluctance to pay for MS and/or MSD analyses, most clients may not know what samples to collect for MS analysis. Clients may not collect sufficient samples, have no knowledge of the native analyte concentrations in the chosen sample, and may not have knowledge of MS spiking levels that are appropriate either for the sample or their project. For procedures with multiple target analytes, spiking levels may be inappropriate for one or more analytes, if samples that contain target analytes are chosen either in the field or by the laboratory. If no project samples are collected for MS analysis, samples from another project used for MS analysis may not be representative of the project samples. When project samples (e.g. surface soil) are chosen, their MS recovery characteristics may be different from those of other samples (e.g. sub-surface soil) in the same batch.

12.10.4 ACTION - If the sample chosen for MS/MSD is inappropriate, notify the client. When additional payments are authorized for re-analysis, re-extract and/or reanalyze the MS/MSD samples after employing cleanup procedures (if applicable), dilution techniques to minimize matrix interference, or using other project samples that appear to be clean, when sufficient quantities are available. Re-spike the sample at a higher level (e.g., at approximately two to four times the sample concentration), then reanalyze the sample based on project-specific requirements. It is the responsibility of the client at all times to select appropriate samples for MS analysis, make payments for additional analyses, and to offer necessary guidance as needed to meet their data quality objectives.

12.10.5 ACTION - If the matrix effect cannot be resolved, discuss the situation in the case narrative. No action may be taken by the laboratory to qualify associated sample data based on MS/MSD recovery data alone. Professional judgment may be used by the user (client) in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

12.10.6 ACTION – No action is taken on MS/MSD recoveries data alone to qualify all associated samples. Since matrix interference could lead to lead to serious analytical problems, the supervisor should be consulted when significant outliers are noted. Document QC outliers for %recovery and RPD on the checklist and on client reports and draw attention to the QC outliers on the case narrative. No action may be taken on MS/MSD recovery data alone either to reanalyze samples and/or to qualify associated samples either by the analyst or by the end user. Professional judgment may be used by the user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

12.11 INTERNAL STANDARD PERFORMANCE – Internal Standard (IS) performance criteria ensures that the GC sensitivity and response is stable during each run.

12.11.1 IS area counts (use mean IS area from ICAL for the %D of IS area) must not vary by more than a factor of 2 (-50% or + 100%) from the associated ICAL/Calibration Verification standard. The internal standard areas for the CCV should not differ by a factor of 2 (-50% or + 100%) from the initial calibration of the same standard.

12.11.2 The retention time of the internal standard must not vary by more than 30 seconds from the associated daily calibration standard.

12.11.3 ACTION – If an internal standard is outside of QC limits of the associated daily standard, then re-analysis is required or a dilution may be required if it appears that interference's occur from target and non-target analytes at high concentrations relative to the ICAL. Do not dilute the sample to the point where all target analytes are non-detect. Professional judgment must be used when dilutions are required for IS criteria.

12.11.4 ACTION – If an internal standard is outside of QC limits of the associated ICAL or CCV standard, correct the problem and re-analyze samples with a valid CCV. If re-analysis has been performed and it is still out, it is of the professional judgment of the analyst to dilute or not to dilute the sample further so as to not to dilute out any target analytes.

12.11.5 ACTION - Report results without qualification describing the QC problem in the case narrative. The user (client) may qualify positive results for compounds quantitated using the affected IS as estimated (J). If a very low IS area is reported, indicating a loss of sensitivity, non-detects may require qualification as invalid (I) or rejected (R). Document recommendation for the client in the case narrative.

12.11.6 ACTION – If an IS retention time varies by more than +/- 30 seconds, the total ion profile must be checked for any false positives or negatives. If a large shift has occurred, then professional judgment must be used as to whether reanalyze or not. See previous section for guidance.

12.12 TARGET COMPOUND IDENTIFICATION – Criteria for GC/ECD qualitative analysis is to minimize the number of erroneous positive or negative identifications of compounds.

12.12.1 Positive compounds must be within +/- 0.06 relative time (RRT) units of the standard RRT. The results should be >MDL or other threshold for reporting positives (e.g. LLR, RL) on both columns.

12.12.2 The RPD for sample amounts from the two columns for identified compounds should be within <40%. Document RPD calculations (See Appendix 6 for details) in Sample Result worksheets (Form-10 equivalents).

12.12.3 ACTION - If the RPD for positive results are <40%, report the result from the primary column without any flag.

12.12.4 ACTION - If the RPD is >40%, report the result from the primary column with a "J" flag. The result from the confirmation column may be reported with a "J" flag at the discretion of the analyst, if interference is suspected for the compound of interest in the primary column.

12.12.5 Professional judgment must be used when applying the criteria for GC/ECD analysis of target analytes. Take into account factors such as matrix effect and column bleed.

13 REFERENCES

13.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8151A, Chlorinated Herbicides by GC using Methylation or Pentafluorobenzylation Derivatization, December 1996.

13.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.

13.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

13.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3500B, Organic Extraction and Sample Preparation, December 1996.

13.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3600C, Cleanup, December 1996.

13.6 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3640A, Gel-Permeation Cleanup, December 1996.

13.7 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3660B, Sulfur Cleanup, December 1996.

13.8 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3580A, Waste Dilution, December 1996.

13.9 EPA Method 515.1, Determination of Chlorinated Acids in Drinking Water Gas Chromatography with Electron Capture Detection, Revision 4.0, 1989, Technical Support center, Office of Ground Water and Drinking Water, U.S. EPA, Cincinnati, OH 45268.

13.10 EPA Method 515.2, Determination of Chlorinated Acids in Drinking Water Using Liquid-Liquid Extraction, Gas Chromatography with Electron Capture Detection, Revision 1.0, 1992, Technical Support center, Office of Ground Water and Drinking Water, U.S. EPA, Cincinnati, OH 45268.

13.11 EPA Method 515.3, Determination of Chlorinated Acids in Drinking Water by Liquid-liquid Microextraction, Derivatization, and Gas Chromatography with Electron Capture Detection, Revision 1.0, July 1996, Technical Support center, Office of Ground Water and Drinking Water, U.S. EPA, Cincinnati, OH 45268.

13.12 EPA Method 515.4, Determination of Chlorinated Acids in Drinking Water by Liquid-liquid Microextraction, Derivatization, and fast Gas Chromatography with Electron Capture Detection, Revision 1.0, April 2000, Technical Support center, Office of Ground Water and Drinking Water, U.S. EPA, Cincinnati, OH 45268.

13.13 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.

13.14 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

13.15 AML Standard Operating Procedures, Q-Water-RC.

13.16 AML Standard Operating Procedures, Q-WasteMgmt-RD.

13.17 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

13.18 AML Standard Operating Procedures, Q-Controlcharts-RC.

Table I

HERBICIDE CALIBRATION STOCK SOLUTION

Compound	Stock Solution Concentration (ug/ml)
2,4-D	100
Dalapon	250
2,4-DB	100
Dicamba	10
Dichlorprop	100
Dinoseb	50
MCPA	10000
MCPP	10000
Silvex (2,4,5-TP)	10
2,4,5-T	10

NOTE: The stock solution is used for calibration.

Table II

HERBICIDE CALIBRATION INTERMEDIATE SOLUTION
and
HERBICIDE INITIAL CALIBRATION SOLUTIONS (HERB-5)

Compound	Intermediate Solution (also Herbicide STD-5) Concentration (ng/ml)
2,4-D	1000
Dalapon	2500
2,4-DB	1000
Dicamba	100
Dichlorprop	1000
Dinoseb	500
MCPA	100000
MCPP	100000
Silvex (2,4,5-TP)	100
2,4,5-T	100

NOTE: The intermediate solution also serves as the high point for the initial calibration (ICAL-HIGH).

Table III

HERBICIDE INITIAL CALIBRATION SOLUTIONS (HERB-4)

Compound	Intermediate Solution Concentration	Intermediate Solution Volume	Hexane Volume	Herbicide STD-4 Concentration
2,4-D	1000 ng/ml	700 uls	300 uls	700 ng/ml
Dalapon	2500 ng/ml	700 uls	300 uls	1750 ng/ml
2,4-DB	1000 ng/ml	700 uls	300 uls	700 ng/ml
Dicamba	100 ng/ml	700 uls	300 uls	70 ng/ml
Dichlorprop	1000 ng/ml	700 uls	300 uls	700 ng/ml
Dinoseb	500 ng/ml	700 uls	300 uls	350 ng/ml
MCPA	10000 ng/ml	700 uls	300 uls	7000 ng/ml
MCPP	10000 ng/ml	700 uls	300 uls	7000 ng/ml
Silvex	100 ng/ml	700 uls	300 uls	70 ng/ml
2,4,5-T	100 ng/ml	700 uls	300 uls	70 ng/ml

NOTE: This solution serves as the mid-high point for the initial calibration (ICAL-MID HIGH).

Table IV

HERBICIDE INITIAL CALIBRATION SOLUTIONS (HERB-3)

Compound	Intermediate Solution Concentration	Intermediate Solution Volume	Hexane Volume	Herbicide STD-3 Concentration
2,4-D	1000 ng/ml	500 uls	500 uls	500 ng/ml
Dalapon	2500 ng/ml	500 uls	500 uls	2250 ng/ml
2,4-DB	1000 ng/ml	500 uls	500 uls	500 ng/ml
Dicamba	100 ng/ml	500 uls	500 uls	50 ng/ml
Dichlorprop	1000 ng/ml	500 uls	500 uls	500 ng/ml
Dinoseb	500 ng/ml	500 uls	500 uls	250 ng/ml
MCPA	10000 ng/ml	500 uls	500 uls	5000 ng/ml
MCPP	10000 ng/ml	500 uls	500 uls	5000 ng/ml
Silvex	100 ng/ml	500 uls	500 uls	50 ng/ml
2,4,5-T	100 ng/ml	500 uls	500 uls	50 ng/ml

NOTE: This solution serves as the mid point for the initial calibration (ICAL-MID). This solution also serves as the calibration verification standard.

Table V

HERBICIDE INITIAL CALIBRATION SOLUTIONS (HERB-2)

Compound	Intermediate Solution Concentration	Intermediate Solution Volume	Hexane Volume	Herbicide STD-2 Concentration
2,4-D	1000 ng/ml	300 uls	700 uls	300 ng/ml
Dalapon	2500 ng/ml	300 uls	700 uls	750 ng/ml
2,4-DB	1000 ng/ml	300 uls	700 uls	300 ng/ml
Dicamba	100 ng/ml	300 uls	700 uls	30 ng/ml
Dichlorprop	1000 ng/ml	300 uls	700 uls	300 ng/ml
Dinoseb	500 ng/ml	300 uls	700 uls	150 ng/ml
MCPA	10000 ng/ml	300 uls	700 uls	3000 ng/ml
MCPP	10000 ng/ml	300 uls	700 uls	3000 ng/ml
Silvex	100 ng/ml	300 uls	700 uls	30 ng/ml
2,4,5-T	100 ng/ml	300 uls	700 uls	30 ng/ml

NOTE: This solution serves as the low-mid point for the initial calibration (ICAL-LOW MID).

Table VI

HERBICIDE INITIAL CALIBRATION SOLUTIONS (HERB-1)

Compound	Intermediate Solution Concentration	Intermediate Solution Volume	Hexane Volume	Herbicide STD-1 Concentration
2,4-D	1000 ng/ml	100 uls	900 uls	100 ng/ml
Dalapon	2500 ng/ml	100 uls	900 uls	250 ng/ml
2,4-DB	1000 ng/ml	100 uls	900 uls	100 ng/ml
Dicamba	100 ng/ml	100 uls	900 uls	10 ng/ml
Dichlorprop	1000 ng/ml	100 uls	900 uls	100 ng/ml
Dinoseb	500 ng/ml	100 uls	900 uls	50 ng/ml
MCPA	10000 ng/ml	100 uls	900 uls	1000 ng/ml
MCPP	10000 ng/ml	100 uls	900 uls	1000 ng/ml
Silvex	100 ng/ml	100 uls	900 uls	10 ng/ml
2,4,5-T	100 ng/ml	100 uls	900 uls	10 ng/ml

NOTE: This solution serves as the low point for the initial calibration (ICAL-LOW).

Table VII

HERBICIDE MS/MSD SPIKING SOLUTIONS

Compound	MS/MSD Spike Soln. Concentration	MS/MSD Spike Soln. Volume (mL)	Aqueous Sample Volume(mL)	Final Sample Extract Volume (mL)	Conc. ng/mL
2,4-D	2500 ng/ml	1-mL	1000-mL	5-mL	500-ng/mL
Dalapon	11250 ng/ml	1-mL	1000-mL	5-mL	2250-ng/mL
2,4-DB	2500 ng/ml	1-mL	1000-mL	5-mL	500-ng/mL
Dicamba	250 ng/ml	1-mL	1000-mL	5-mL	50-ng/mL
Dichlorprop	2500 ng/ml	1-mL	1000-mL	5-mL	500-ng/mL
Dinoseb	1250 ng/ml	1-mL	1000-mL	5-mL	250-ng/mL
MCPA	25000 ng/ml	1-mL	1000-mL	5-mL	5000-ng/mL
MCPP	25000 ng/ml	1-mL	1000-mL	5-mL	5000-ng/mL
Silvex	250 ng/ml	1-mL	1000-mL	5-mL	50-ng/mL
2,4,5-T	250 ng/ml	1-mL	1000-mL	5-mL	50-ng/mL

Table VIII

HERBICIDE LCS/LCSD SPIKING SOLUTIONS

Compound	LCS/LCSD Spike Soln. Concentration	LCS/LCSD Spike Soln. Volume (mL)	Aqueous Sample Volume(mL)	Final Sample Extract Volume (mL)	Conc. ng/mL
2,4-D	2500 ng/ml	1-mL	1000-mL	5-mL	500-ng/mL
Dalapon	11250 ng/ml	1-mL	1000-mL	5-mL	2250-ng/mL
2,4-DB	2500 ng/ml	1-mL	1000-mL	5-mL	500-ng/mL
Dicamba	250 ng/ml	1-mL	1000-mL	5-mL	50-ng/mL
Dichlorprop	2500 ng/ml	1-mL	1000-mL	5-mL	500-ng/mL
Dinoseb	1250 ng/ml	1-mL	1000-mL	5-mL	250-ng/mL
MCPA	25000 ng/ml	1-mL	1000-mL	5-mL	5000-ng/mL
MCPP	25000 ng/ml	1-mL	1000-mL	5-mL	5000-ng/mL
Silvex	250 ng/ml	1-mL	1000-mL	5-mL	50-ng/mL
2,4,5-T	250 ng/ml	1-mL	1000-mL	5-mL	50-ng/mL

Table IX
Aqueous Sample MDL, LLR, MQL and QC Limits for LCS and MS

Chlorinated Herbicides Analysis													
Analytical Management Laboratories													
Preparation Method:	8151A						Derivatization reagent:				Diazomethane		
Analysis Method:	8151A						CAL standards:				Methyl esters		
Matrix: water	**MDL**	**MDL**	**MDL**	**MDL**	**LLR**	**MQL**	**Ratio (RQ)**		**DoD QSM LCS & MS Control Limits**		**DoD QSM LCS & MS ME Limits**		**RPD**
Compound	Spike	DB-5	DB-1701	Report	Report	Report	QL/DL	flag	Lower	Upper	Lower	Upper	Limit
	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L			**%R**	**%R**	**%R**	**%R**	%
Dalapon	0.375	0.066	0.039	**0.066**	0.132	**0.625**	9.5		40	110	40	110	50
Dicamba	0.015	0.021	0.031	**0.031**	0.061	**0.100**	3.3		60	110	60	110	50
MCPP	15	22.83	16.30	**22.83**	45.65	**100**	4.4		50	150	50	150	50
MCPA	15	5.02	17.04	**17.04**	34.08	**100**	5.9		60	145	60	145	50
Dichloroprop	0.15	0.042	0.075	**0.075**	0.15	**0.250**	3.3		70	120	70	120	50
2,4-D	0.15	0.035	0.052	**0.052**	0.103	**0.250**	4.8		35	115	35	115	50
Silvex (2,4,5-TP)	0.015	0.005	0.003	**0.005**	0.01	**0.025**	4.9		50	115	50	115	50
2,4,5-T	0.015	0.004	0.003	**0.004**	0.008	**0.025**	6.4		35	110	35	110	50
Dinoseb	0.075	0.015	0.006	**0.015**	0.031	**0.125**	8.1		20	95	20	95	50
2,4-DB	0.15	0.049	0.030	**0.049**	0.099	**0.250**	5.1		45	130	45	130	50
Surrogate:													
DCAA									50	150			
Criteria for the ratio (RD) of mean amount found to MDL: >1 but <5 for water and for MQL/MDL ratios (RQ) to be >3.													
Default QC limits (50% to 150%) are used for MCPP.													
RPD limits are those described for pesticides (SW 8081) in USACE EM200-1-3, Appendix I.													
Default AML QC limits are used for surrogate recoveries.													
The following criteria will apply for marginal exceedances:													
Number of allowed ME analytes per total analytes: 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).													
NOTE: Marginal exceedance concept is not applicable to herbicides analysis. Control limits and ME limits are identical.													
NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.													

Table X
Soil/sediment Sample MDL, LLR, MQL and QC Limits for LCS and MS

Chlorinated Herbicides Analysis

Analytical Management Laboratories

Preparation Method:	8151A						Derivatization reagent:				Diazomethane		
Analysis Method:	8151A						CAL standards:				Methyl esters		
Matrix: Soil	**MDL**	**MDL**	**MDL**	**MDL**	**LLR**	**MQL**	**Ratio (RQ)**		**DoD QSM LCS & MS Control Limits**		**DoD QSM LCS & MS ME Limits**		**RPD**
Compound	Spike	DB-5	DB-1701	Report	Report	Report	QL/DL	flag	Lower	Upper	Lower	Upper	Limit
	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg	ug/kg			**%R**	**%R**	**%R**	**%R**	%
Dalapon	12.5	2.62	0.90	**2.62**	5.23	**20.8**	8.0		40	110	40	110	50
Dicamba	0.5	1.03	0.66	**1.03**	2.06	**3.33**	3.2		55	110	55	110	50
MCPP	500	633	774	**774**	1549	**3333**	4.3		50	150	50	150	50
MCPA	500	162	251	**251**	502	**3333**	13.3		60	145	60	145	50
Dichloroprop	5	0.97	2.18	**2.18**	4.35	**8.33**	3.8		75	140	75	140	50
2,4-D	5	1.21	1.70	**1.70**	3.41	**8.33**	4.9		35	145	35	145	50
Silvex (2,4,5-TP)	0.5	0.07	0.054	**0.068**	0.135	**0.833**	12.3		45	125	45	125	50
2,4,5-T	0.5	0.06	0.074	**0.074**	0.147	**0.833**	11.3		45	135	45	135	50
Dinoseb	2.5	0.36	0.24	**0.363**	0.726	**8.33**	23.0		5	130	5	130	50
2,4-DB	5	1.08	0.91	**1.08**	2.16	**4.17**	3.9		50	155	50	155	50
Surrogate:													
DCAA									50	150			

Criteria for the ratio (RD) of mean amount found to MDL: >1 but <10 for soil and for MQL/MDL ratios (RQ) to be >3.

Default QC limits (50% to 150%) are used for MCPP. DoD QSM limits are available for 2,4-D, 2,4-DB, 2,4,5-T, 2,4,5-TP, Dicamba, Dinoseb, and Dichloroprop in solid matrices. The aqueous DoD QSM limits are used as interim default limits for soil matrix.

RPD limits are those described for pesticides (SW 8081) in USACE EM200-1-3, Appendix I.

Default AML QC limits are used for surrogate recoveries.

The following criteria will apply for marginal exceedances:

Number of allowed ME analytes per total analytes: 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).

NOTE: Marginal exceedance concept is not applicable to herbicides analysis. Control limits and ME limits are identical.

NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.

Table XI
List of Appendices

Appendix Number	Appendix Topic
1	Diazomethane Preparation
2	Initial Calibration (ICAL) Summary (Form-6 equivalents) Instrument Printouts
3	Calibration Verification (ICV and CCV) Summary (Form-7 equivalents) Instrument Printouts and Standard Chromatograms
4	Retention Time Window Summary
5	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
6	Method Detection Limit (MDL) Studies Summary
7	Sample Reports (Form-1 equivalents)
8	QC Association Forms (Form-4 equivalents) Sample Preparation Logsheets Instrument Run Log
9	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
10	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
11	Surrogate Recovery Reports (Form-2 equivalents)
12	Sample Results Worksheet (Form-10 equivalents)
13	DoD QSM Method Quality Objectives for Method 8151 AFCEE MQOs for Method 8151

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

INSTRUCTIONS FOR HANDLING AND PROCESSING SAMPLES COLLECTED IN VOA VIALS FOR MADEP-VPH ANALYSIS

Soil samples collected for Volatile Petroleum Hydrocarbons (VPH) analysis in accordance with Massachusetts Department of Environmental Protection (MADEP) should be contained in two 40-mL VOA vials and one cut-off syringe for each sample. The tare weight of each vial, which includes the cap, preservative (15-mL methanol), and the sample label is recorded on the label using indelible ink. Weight is recorded to two decimal places (four significant numbers) by AML. The weight of the syringe is not recorded.

Instructions to Field Personnel:

1. Do not attach additional labels to any of the 40-mL vials. The labels will add additional weight (0.3 to 0.7-g depending on the label) to the vial. <u>If you attach any label to the vial, you must re-weigh the vial in the field before and after adding the soil sample and record the weights or the sample weight only on the new label.</u>
2. You may attach a label to the syringe. The sample contained in the syringe is used for the determination of percent solids. Attach additional labels containing detailed sample information (project name, sample location, field sample number, date, time, sampler, etc.) to the bubble-wrap pouch that contains the 40-mL vials and the syringe and seal the pouch with transparent tape.
3. Label each sample vial with the field sample number using indelible ink.
4. The vials contain a mark at 25-mL volume to indicate the level of methanol that should exist when the required quantity of soil (15-g) has been added. Using the cut-off syringe provided, add approximately 15-g soil (See Item #5 on the following page for additional guidance) to each vial without splashing and/or spilling the preservative. For most soils, 8 to 10-mL of methanol is displaced for 15-g soil (depending on moisture content and density of soil). The soil sample in the vial should be completely covered by methanol. Do <u>NOT</u> leave the vials containing methanol open for extended periods of time to minimize solvent evaporation.
5. The outside of the vials and the caps should be clean. Dirt will add additional weight to the vials, which does not represent sample weight used in the analysis.
6. The threads should be clean and free of dirt. Otherwise, the vials may not seal properly and the volatile organic compounds may escape from the vial.
7. If possible, weigh the vial in the field to two decimal places and record it directly on the vial. Ship the samples as quickly as possible to the laboratory. When the weight of vials is determined accurately in the field, the laboratory will make these measurements.
8. See Item #3 and #8 on the following page for information on trip blank.

Instructions to Laboratory Sample Receiving Personnel:

1. After initial inspection, record laboratory sample number on each vial and the syringe using indelible ink.
2. Allow the sample vials to reach room temperature before weighing each vial to two decimal places (to four significant numbers) and recording them on the vial using indelible ink. Do NOT open any of the vials. The sample custodian may attach additional stick-on labels to the vials after recording the sample weight.
3. AML personnel should complete Form-C (from the 5035 Method) if samples are preserved in the field. Other laboratories (e.g. QA Lab) should use their own forms and procedures to collect and record the information.
4. Check and tighten the cap on the vials before storing them in the VOA refrigerator.

MADEP-VPH SAMPLE COLLECTION

This option will be used by AML as the default option for VHP sample collection and analysis in accordance with MADEP, when other options are not specified in the FSP or SAP. Balances may be useful but not required for sample collection in the field for this option. Under this option, samples are collected into vials containing a known amount of methanol, chilled to 4°C and shipped as soon as possible. Upon receipt, the samples are examined, weighed, logged in and stored frozen at 4°C until analysis.

1. Make a mark at 25-mL on 40-mL VOA vials with indelible ink. Pipette out 15-mL of purge and trap grade or an equivalent grade methanol. Attach a label to each vial. Determine the weight (g) of vial containing methanol to two decimal places and record it on the label using indelible ink.
2. For each soil or sediment sample to be collected for MADEP-VPH analysis, assemble a kit, which consists of the following: two pre-weighed and labeled 40-ml VOA vials, one EasyDraw Syringe®, and a foam pouch.
3. For trip blank, assemble a kit, which consists of the following: two pre-weighed and labeled 40-ml VOA vials and a foam pouch.
4. For each sampling event, assemble a kit, consisting of the following: one Powerstop Handle® syringe holder; cooler(s) of appropriate size; trip blank(s), and zip-lock bag containing chain of custody forms, sample labels, custody seal, etc.
5. The syringe holder will have settings for collecting 5-gram, 10-g or 13-gram samples for different analyses. Remove cap from the syringe. Insert the syringe into one of the 13-g position. Push the syringe into the representative soil being sampled until the soil column inside the syringe has forced the plunger to the stop point (top of the syringe holder). Remove the syringe from the holder, wipe the exterior of the syringe with a paper towel, insert syringe into one of the open pre-weighed 40-ml vials and eject sample into it. Avoid getting dirt on the threads of the 40-ml vial. Wipe the vial threads if necessary, cap vial immediately, and store at 4°C. Alternatively, 15-g may be collected using a two-step procedure that involves using the syringe in 5-g and 10-g positions in sequence.
6. Collect an additional 40-mL vial for each sample. Use the same syringe for collecting additional vials.
7. Insert the syringe into the lowest (13-g) position. Collect an additional aliquot for the determination of percent solids and cap syringe.
8. The trip blank is methanol, which should be the same lot of methanol as used for soil sample collection. Do not add anything (soil or reagent water) in the field or the laboratory to the vials that are labeled as "Trip Blank".
9. Upon receipt at the laboratory, the sample custodian will assign a laboratory sample number for each sample and labels such as A and B to the 40-ml vials and document them in modified Form-C. The sample custodian will copy into Form-C the tare weight (VP) of each vial that has been pre-determined and recorded on the vials. The sample custodian will also copy the field-determined weight (FW) when it is available.
10. The sample custodian will determine the weight of the vial plus sample (LW) to two decimal places in the laboratory and record it on Form-C. When available, the difference between the field and lab weights (FW-LW) will be used to evaluation sample integrity. When applicable, the sample weight is the difference between the field weight (FW) and the tare weight (VP). Otherwise, the sample weight is the difference between the lab weight (LW) and the tare weight (VP).
11. Immediately before analysis, the analyst will allow the vials to warm up to room temperature and perform analysis as described in the respective analytical method.

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(Mod. FORM-C) MADEP VPH Soil Sample Collection and Preservation Record

CLIENT PROJECT & NO: ____ AML WORK ORDER NO: _______________

Date of sample collection:					Date (log-in): **(lab use only)**				
Field Sample Number (field use only)	Vial Number	Preservative M = Methanol (15-mL)	Weight (VP, g) Of Vial (V) + preservative (P)	Weight (FW, g) V P + sample (S) (optional field use)	Weight (LW, g) VP + Sample (S) **(lab use only)**	Wt. Diff. (FW-LW) (optional)	Sample (S) Weight (g) (LW-VP)	Lab. Sample Number **(lab use only)**	Comments **(lab use only)**

STANDARD OPERATING PROCEDURES

No. O-PAH-GCMS-SIM-RC

Polynuclear Aromatic Hydrocarbons (PAHs or PNAs) by Gas Chromatography/Mass Spectrometry (GC/MS) in the Selected Ion Monitoring (SIM) Mode (EPA 625 and SW-846 8270D)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Polynuclear Aromatic Hydrocarbons (PAHs or PNAs) by Gas Chromatography/Mass Spectrometry (GC/MS) in the Selected Ion Monitoring (SIM) Mode (EPA 625 and SW-846 8270D)

1.0 SCOPE AND APPLICATION

1.1 This Analytical Management Laboratories (AML) standard operating procedure (SOP) is based on EPA SW-846 Method 8270C/8270D, EPA Method 625, and EPA contract laboratory program (CLP) methods OLM02.0, OLM03.0, and OLM04.0.

1.2 This SOP is used to determine polynuclear aromatic hydrocarbons (PAHs or PNAs) in a variety of matrices including water, air, soil and waste. Water samples will include groundwater, surface water, drinking water, and wastewater.

1.3 Sample preparation procedures contained in this SOP are based on EPA SW-846 and other EPA methods that are referenced in this SOP. Words and acronyms such as method and SOP are used interchangeably in this document.

1.4 This SOP can be used to quantitate most PAHs that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with DB5. ***Table 1*** lists the analytes or compounds and their retention times.

1.5 The practical quantitation limits (MQLs) for each individual compound is instrument dependent and highly matrix dependent. The instrument employed at AM Laboratories is a standard Agilent (formerly HP) quadrupole 5973 mass selective detector (MSD). See ***Tables 2 and 3*** for MQLs that are generally achievable.

1.6 This SOP uses specialized gas chromatography/mass spectrometry (GC/MS) procedures. This SOP is restricted to experienced analysts in the use of GC/MS theories and skilled in the interpretation of mass spectra and their use as a quantitative tool. Knowledge of and/or experience with other selected ion monitoring (SIM) methods such those based on EPA 613, 1613, 8280 and 8290 is desirable.

2.0 SUMMARY OF METHOD

2.1 This SOP provides extraction and gas chromatographic conditions for the analysis of polynuclear aromatic hydrocarbon compounds. Samples are extracted using appropriate matrix-specific procedures, concentrated to 1.0-mL, and analyzed by GC/MS. Two procedures are described:

2.1.1 Procedure -A involves sample preparation that is identical to that for SVOCs analysis. The SVOCs extracts will be analyzed using GC/MS in the SIM mode and a calibration procedure that will result in quantitative analysis for PAHs at sub-µg/L levels. The same extract is also used for quantitative analysis in the full scan mode for the broad spectrum of compounds. This procedure combines the versatility of the full scan analysis and the low detection capabilities of the SIM mode.

2.1.2 Procedure -B is specific to PAHs analysis. Solid phase extraction cartridges (EPA 550.1 and SW-846 3535) are used for the extraction of aqueous samples, which may not be applicable to the full spectrum analysis of SVOCs. Samples are spiked with

base neutral (BN) surrogates only at low concentrations. Associated QC samples (LCS/LCSD, and MS/MSD) are spiked only with PAH target compounds at a much lower concentrations. The internal standards are also added at a much lower concentration.

2.1.3 The Laboratory will make the decision to choose the correct analytical procedure to meet data quality objectives for specific projects.

2.2 The GC/MS is operated in the selected ion monitoring (SIM) mode for both procedures (A and B) for PAH analysis. Therefore, instrument sensitivities are identical for both procedures.

3.0 INTERFERENCES

3.1 Interferences can be caused by a number of sources in the extraction and analyses stages. Quality control measures, such as method blanks, solvent blanks and matrix spikes can be used to determine the interference.

3.2 Contamination may occur when a high-concentration and a low concentration sample are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. A high concentrated sample should be followed by the analysis of solvent to check for possible cross contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph - Hewlett Packard (Agilent) 6890 Series II GCs are used. The systems are complete with Agilent 7673/7673A autosamplers, with a temperature-programmable GC for splitless injection. The system includes all accessories, including syringes, analytical columns and gasses. The column flow rate remains constant throughout temperature program operation. The capillary column is directly coupled to the source.

4.2 Gas chromatographic column - A 30 meter x 0.25-mm ID capillary column coated with DB-5 (J&W Scientific) is employed. The film thickness is 0.25 um. An equivalent column may be used.

4.3 Mass Spectrometer - Hewlett Packard (Agilent) 5973 Mass Selective Detectors are used. It is capable of scanning from 35 to 800 AMU every 1-second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The instrument can be operated in the full scan or selected ion monitoring (SIM) mode.

4.4 GC/MS interface is directly coupled by inserting the column into the mass spectrometer.

4.5 Data system - Hewlett Packard's Enviroquant is used to allow continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the GC program. The software allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). The software also allows integrating the abundances in any EICP between specified times or scan-number limits.

4.6 Microsyringes - 10, 25, 100, 250, 500 and 1000 uLs.

4.7 Balance - Analytical, 0.0001 grams and top loading, 0.1 grams.

4.8 Disposable pipettes - Pasteur

4.9 Test tubes - 5 ml to 8 ml, with Teflon lined screw caps.
4.10 Separatory funnels - 2000 mL
4.11 Kuderna-Danish (K-D) apparatus
- 4.11.1 10- to 15-ml concentrator tubes
- 4.11.2 Evaporation flask (500-ml), attached to concentrator tubes
- 4.11.3 Three ball macro Snyder column
- 4.11.4 Clamps

4.12 Boiling Chips - solvent extracted
4.13 Heated water bath
4.14 Microsyringes - 10-, 50-, 100-, 500-, and 1000-uL.
4.15 Tekmar Sonic Disrupter
4.16 Glass wool
4.17 Stainless steel table spoons
4.18 SPE extraction equipment:
- 4.18.1 SPE manifold (Supelco) with 12-positions for sample extraction, vacuum control and reservoir for spent water sample collection (Kontes Ultraware).
- 4.18.2 Vacuum pump (Gast Model 2Z866)
- 4.18.3 Supelco SPE extraction cartridges with C-18 phase; Waters Porapak® R_{DX} C-18 SPE cartridge (Part # WAT047220, reserve lot # W1023B3 or equivalent for one-year supply)
- 4.18.4 Sep-Pak adapter for Waters Porapak® R_{DX} C-18 SPE cartridges
- 4.18.5 Sep-Pak reservoirs for Waters Porapak® R_{DX} C-18 SPE cartridges

4.19 Culture tubes, 13 x 100 mm (Fisher)
4.20 Eppendorf pipettes, 100-1000 µL and 10-100 µL

5.0 REAGENTS

5.1 It is assumed that all inorganic/organic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where available. Any other reagents must demonstrate high purity without lessening the accuracy of the determination.
5.2 Organic free reagent water - All references to water in this method will be assumed to be organic free reagent water, except when referring to field samples.
5.3 Sulfuric acid solution
- 5.3.1 Slowly add 50 mL of sulfuric acid to 50 mL of water (1:1 v/v)

5.4 Solvents
- 5.4.1 Acetone – GC grade
- 5.4.2 Hexane – GC grade
- 5.4.3 Methylene chloride - GC grade

5.5 Primary Stock PAH Standard Solution - This is purchased from Ultra Scientific (catalog number US-106N) at a concentration of 2000-µg/mL. Stock standard solutions expire one year from the date of preparation. Replace sooner if signs of degradation are observed.

5.6 Intermediate PAH Standard Solution - This is prepared at a concentration of 25-µg/mL by diluting 125-µL of the 2000-µg/mL stock solution (Section 5.5) to 10-mL with methylene chloride. This standard is replaced every six months or sooner depending on the extent that they are used.

5.7 Semivolatiles GC/MS tuning standard solution, which contains DFTPP, 4, 4'-DDT, PCP, & Benzidine, each at a concentration of 1000-µg/mL is purchased from Ultra (cat # GCM-150). Prepare a 50-µg/mL standard by diluting 1.0-mL of the Ultra Scientific standard to 20-mL with methylene chloride. The tuning standard solution expires one year from the date of preparation. Replace sooner if signs of degradation are observed.

5.8 Surrogate Stock Standard - The surrogate employed depends on the procedure employed, which are shown in Table 6:

5.8.1 Procedure A - The extract will be used for full scan SVOCs analysis and SIM mode PAHs analysis. Surrogate spike levels are higher to accommodate SVOCs analysis. The laboratory employs six surrogates (3-BN and 3-A) as shown in Table 1. The surrogate compounds are phenol-d6, 2-fluorophenol, 2, 4, 6-tribromophenol, nitrobenzene-d5, 2-fluorobiphenyl and p-terphenyl-d14. Surrogates solution standards are purchased from Ultra Scientific (Catalog number ISM-336x). Stock standard solutions expire one year from the date of preparation. Replace sooner if signs of degradation are observed. Spike 0.5-mL of the surrogate solution into each sample including all QC.

5.8.2 Procedure B - The laboratory employs three base neutral surrogates as shown in Table 6. The surrogate compounds are nitrobenzene-d5, 2-flurorbiphenyl and p-terphenyl-d14. Surrogates Stock Standards (1000-µg/mL) are purchased from Ultra Scientific (Catalog number ISM-280N). Stock standard solutions expire one year from the date of preparation. Replace sooner if signs of degradation are observed.

5.9 Surrogate spiking Solution - Prepare for the two procedures as follows:

5.9.1 Procedure A - Spike 0.5-mL of the stock surrogate solution from the vendor into each sample including all QC samples. Dilution is not required. The final concentration in the sample extract (1-mL) will be 50-µg/mL for the BN surrogates and 75-µg/mL for acid surrogates.

5.9.2 Procedure B - Dilute 400-µL of the Ultra stock (Section 5.8.2) to 100-mL to prepare spiking solution at 4-µg/mL. Spike 0.5-mL of this solution into each sample including all QC samples. The final concentration in the sample extract (1-mL) will be 2-µg/mL for the BN surrogates.

5.10 Internal Standards Stock Solution (ISSS) - A stock solution of six analytes (acenaphthene-d_{10}, chrysene-d_{12}, 1, 4-dichlorobenzene-d_4, naphthalene-d_8, perylene-d_{12}, and phenanthrene-d_{10}) at a concentration of 4000-µg/mL is purchased from Ultra (catalogue # US-108N).

5.10.1 Procedure A - Spike the final extracts (1-mL) with 10-µL of the ISSS (Section 5.10) including all QC sample extracts. The final concentration of the internal standards will be 40-µg/mL.

5.10.2 Procedure B - Dilute 62.5-µL of the Ultra stock (Section 5.10) to 1-mL methylene chloride to prepare IS spiking solution at 250-µg/mL. Spike 10-µL of this solution into each sample extract (1-mL) including all QC sample extracts. The final concentration in the sample extract (1-mL) will be 2.5-µg/mL.

5.11 PAH Initial Calibration (ICAL) Solutions - These working standards are prepared daily from the Intermediate PAH Standard Solution (Section 5.6) the surrogate stock solution (Section 5.8.1 or 5.8.2) and the internal standard stock solution (Section 5.10.1 or 5.10.2). The working standards are prepared at a minimum of five concentration levels for each parameter of interest. When six levels of standards are prepared, the analyst may use all six points or drop one point, if curvature is suspected. The analyst can drop either the lowest point or the highest point (depending on project requirements) but not any of the middle points. These standards are replaced every six months or sooner depending on the extent that they are used.

5.11.1 Procedure A - Prepare 2-mL of the highest concentration standard containing PAHs at 5.0-µg/mL and the surrogates at 50-µg/mL. Dilute 400-µL of the 25-µg/mL PAH standard (Section 5.6) and 1000-µL of the surrogate standard (Section 5.8.1) to 2-mL in methylene chloride.

5.11.1.1 Add 10-µL of the IS solution (Section 5.10.1) to 1-mL of the PAH surrogate mixture (Section 5.11.1).

5.11.1.2 Dilute 10-µL of the IS solution (Section 5.10.1) and 0.5-mL of the PAH surrogate mixture (Section 5.11.1) to 1-mL with methylene chloride.

5.11.1.3 Dilute 10-µL of the IS solution (Section 5.10.1) and 0.2-mL of the PAH surrogate mixture (Section 5.11.1) to 1-mL with methylene chloride.

5.11.1.4 Dilute 10-µL of the IS solution (Section 5.10.1) and 0.1-mL of the PAH surrogate mixture (Section 5.11.1) to 1-mL with methylene chloride.

5.11.1.5 Dilute 10-µL of the IS solution (Section 5.10.1) and 0.05-mL of the PAH surrogate mixture (Section 5.11.1) to 1-mL with methylene chloride.

5.11.2 Procedure B - Prepare 2-mL of the highest concentration standard containing PAHs at 5.0-µg/mL and the surrogates at 2-µg/mL. Dilute 400-µL of the 25-µg/mL PAH standard (Section 5.6) and 1000-µL of the surrogate standard (Section 5.9.2) to 2-mL in methylene chloride.

5.11.2.1 Add 10-µL of the IS solution (Section 5.10.2) to 1-mL of the PAH surrogate mixture (Section 5.11.2).

5.11.2.2 Dilute 10-µL of the IS solution (Section 5.10.2) and 0.5-mL of the PAH surrogate mixture (Section 5.11.2) to 1-mL with methylene chloride.

5.11.2.3 Dilute 10-µL of the IS solution (Section 5.10.2) and 0.2-mL of the PAH surrogate mixture (Section 5.11.2) to 1-mL with methylene chloride.

5.11.2.4 Dilute 10-µL of the IS solution (Section 5.10.2) and 0.1-mL of the PAH surrogate mixture (Section 5.11.2) to 1-mL with methylene chloride.

5.11.2.5 Dilute 10-µL of the IS solution (Section 5.10.2) and 0.05-mL of the PAH surrogate mixture (Section 5.11.2) to 1-mL with methylene chloride.

5.12 Laboratory Control Sample (LCS) and Matrix Spike (MS) spiking solution

5.12.1 Procedure A - Spiking solution for this procedure is prepared from the Stock Standard Solution used with SVOCs analysis (Catalogue No. SVM-8270 from Ultra Scientific). Dilute 2.5-mL of the stock solution (1000-µg/mL) to 50-mL with acetone to prepare the spiking solution at a concentration of 50-µg/mL. Spike 1-mL into LCS,

LCSD, MS and MSD samples. The spiking solution expires in three months from the date of preparation. Replace sooner if signs of degradation are observed.

5.12.2 Procedure B - Spiking solution for this procedure is prepared from the Intermediate PAH Stock Standard (Section 5.6). Dilute 5-mL of this solution (25-µg/mL) to 50-mL with acetone to prepare the spiking solution at a concentration of 2.5-µg/mL. Spike 1-mL into LCS, LCSD, MS and MSD samples. The spiking solution expires in three months from the date of preparation. Replace sooner if signs of degradation are observed.

5.13 Method Blank (MB) Samples - These are prepared from controlled matrices for procedure A and B.

5.13.1 Aqueous samples - Use 1-L of reagent water.

5.13.2 Soil or sediment samples - Use an appropriate amount (30 or 10-g) of sodium sulfate instead of the actual sample.

5.14 Laboratory Control Samples (LCS and LCSD) - These are prepared by spiking two additional aliquots of controlled matrices prepared as described in Section 5.14.1 with the spiking solution described above (section 5.12) for procedure A and B.

5.15 Matrix Spike Samples (MS and MSD) - These are prepared by spiking two additional aliquots of the actual aqueous or soil sample with the spiking solution described above (section 5.12) for procedure A and B.

5.16 Sodium Hydroxide Solution (10N) - Dissolve 40 grams of sodium hydroxide solution into 100 mLs of reagent water.

5.17 Sodium sulfate - (granular, anhydrous), Na_2SO_4.

5.18 Second Source PAH Standard - The second source standard is used to verify the accuracy of the initial calibration performed using the primary standard.

5.18.1 Stock Standard Solution - This is purchased from Supelco (catalog number 48755-U) at a concentration of 200-µg/mL. The stock standard solution expires one year from the date of preparation. Replace sooner if signs of degradation are observed.

5.18.2 Intermediate Standard Solution - This is prepared at a concentration of 25-µg/mL by diluting 250-µL of the 200-µg/mL stock solution (Section 5.18.1) to 2-mL with methylene chloride. This standard is replaced every six months or sooner depending on the extent that they are used.

5.18.3 Initial Calibration Verification (ICV) Standard - Prepare at mid-level as described for the mid-level ICAL standard (see Section 5.11.1.3 or 5.11.2.3) using the second source PAH standard (Section 5.18.2) and the same source of surrogate and internal standards as for the ICAL solutions.

5.19 Continuing Calibration Verification (CCV) Standard - Prepare a mid-level standard as described in section 5.11.1.3 or 5.11.2.3.

5.20 SVOCs standard - This standard is used to establish retention times for the surrogates, internal standards, and target compounds to make adjustments to the SIM tables and their switching times. Prepare this standard by spiking 10-µL of the 4000-µg/mL internal standard solution (Section 5.10) and 50-µL of the 1000-µg/mL surrogate standard (Section 5.8.2) to 1.0-mL of the LCS/MS spiking solution used for Procedure-A (Section 5.12.1).

6.0 SAMPLE PRESERVATION HANDLING & STORAGE

6.1 One liter amber glass bottles with Teflon lined screw caps may be used for collecting aqueous samples in accordance with EPA SW-846 method 8270C. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 2 bottles for each water sample to be collected. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. See project specific field sampling plan (FSP) for sample collection and preservation instructions. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.

6.2 One 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples in accordance with EPA SW-846 Method 8270C. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each solid sample. Quality control samples such as MS and MSD should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. These samples may be shared with samples collected for other analyses such as chlorinated pesticides (8081A), PCBs (8082) diesel (DRO, 8015-mod.) and other pesticides (8140, etc.).

6.3 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Analytical data generated outside of the specified holding times must be considered to be minimum values only. Such data may be used to demonstrate that a waste is hazardous where it shows the concentration of a constituent to be above the regulatory threshold but cannot be used to demonstrate that a waste is not hazardous. The holding time for the extraction of aqueous samples, which includes TCLP leachates is 7 days from the date of sample collection or TCLP leachate preparation. The holding times for the extraction of soil, sediment and waste samples, which includes TCLP leachate preparation is 14 days from the date of sample collection. The holding time for the instrumental analysis of sample extracts is 40 days from the date of aqueous or solid sample extraction.

7.0 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control contain a number of regulated compounds at high concentrations. The toxicity of these compounds present in such complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Aqueous and soil samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters after the solvent(s) have been allowed to evaporate in a hood.

9.0 SAMPLE EXTRACTION PROCEDURE

Determine the procedure to be followed (A or B). Use surrogate, spiking and internal standard solution that is appropriate to each procedure. Steps described below are identical for procedures A and B.

9.1 Preparation of Sediment and Soil Samples by Sonication

9.1.1 Add 30-grams of soil to a 500-mL beaker. Record the exact weight in the organic Extraction logbook. Add 60-g of sodium sulfate (additional amounts may be needed to dry wet soil samples) and mix well with a spoon. Add 0.5-mL of surrogate solution to all the samples (including QC) and 1-mL of the appropriate spiking solution to LCS/LCSD & MS/MSD samples.

9.1.2 Add 50- to 100-mL of the solvent mixture (methylene chloride: acetone 90:10) to the beaker and mix the contents with a spoon for 1 minute. Sonicate the soil/methylene chloride mixture for 3 minutes ensuring that the soil is free flowing and not in clumps. Decant the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus.

9.1.3 Extract the sample twice more with 50- to 100-mLs of methylene chloride/acetone solvent mixture. The solution should be sonicated for 3minutes each time. Decant the solution in to the same K-D as in the previous step.

9.1.4 Add two boiling chips to the K-D flask and attach a three-ball Snyder column. Prewet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1- to 5-ml, remove the K-D flask and allow to cool for at least 10 minutes.

9.1.5 Concentrate the extract by nitrogen blow-down to 1.0-mL. Proceed to analyze by GC/MS.

9.2 Preparation of Soil samples by Soxhlet. The procedure described below employs the Soxtec equipment. See also AML SOP on SVOCs for detailed procedures for sample preparation using IKA automated Soxhlet equipment (new).

9.2.1 Weight out 10-g of soil. Dry it with 20-g sodium sulfate. Add the entire contents into a fiber glass thimble and place thimble onto a Soxtec unit.

9.2.2 Add 0.5-mL of surrogate solution to all the samples (including QC) and 1-mL of the appropriate spiking solution to LCS/LCSD & MS/MSD samples.

9.2.3 Add 60-mL of dichloromethane and set temperature program to methylene chloride (NOTE: Use program 1 or follow instructions in the manual).

9.2.4 The water recirculator should be set a temperature less than 4°C. Let solvent recirculate for 18 hours.

9.2.5 Collect the extract and perform volume reduction and solvent exchange as described in Section 9.1.4 and 9.1.5.

9.3 Preparation of Water Samples by liquid-liquid extraction

9.3.1 Using a black marker, mark the meniscus of the water in the sample container. Pour the entire contents of the container into a 2-liter separatory funnel. Rinse the sample container with 3 aliquots of 20-mL methylene chloride and place the rinse in the separatory funnel. Add distilled water to the sample container up to the marked meniscus. Pour the distilled water into a 1-liter graduated cylinder. Record this measured volume in the SVOC or PAH (as appropriate) Extraction logbook.

9.3.2 An alternate procedure, which may be employed to measure out water samples, is described below. Using a 1-Liter graduated cylinder, measure out 1.00 ± 0.005 Liters (or lower volumes as low as 100-mL for high concentration samples) of the sample, and transfer it to a 2000-mL separatory funnel. Record the exact volume in the organic Extraction Log Book.

9.3.3 Using pH indicating paper (0-14) record the pH of the sample. This is accomplished by dipping the tip of a Pasteur pipette into the water and placing the droplets of water on the pH paper. Never place the pH paper into the water sample. Add 0.5-mL of surrogate solution to all of the samples (including QC) and 1-ml of the appropriate spiking solution to the LCS/LCSD & MS/MSD. Adjust the pH to 7±2 (5 to 9) with sodium hydroxide (Section 5.16) or 50/50 sulfuric acid solution (Section 5.3).

9.3.4 Add 60-mLs of methylene chloride to the separatory funnel and shake for 2 minutes. Take care to vent the separatory funnel to relieve excess pressure. Allow the organic layer to separate from the water layer for a minimum of 10 minutes. Drain the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus. Repeat the extraction 2 more times (total 3 extractions) using 60-mLs of methylene chloride. Combine the extracts into a single KD vessel.

9.3.5 Add two boiling chips to the K-D flask and attach a three-ball Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1 to 5-mL, remove the K-D flask and allow cooling for at least 10 minutes.

9.3.6 Concentrate the extract by nitrogen blow-down to 1.0-mL. Proceed to analyze by GC/MS.

9.4 Extraction of Aqueous Samples by SPE Method

9.4.1 Perform extractions using Supelco SPE vacuum manifold.

9.4.2 Condition the SPE tubes with 2 to 5-ml of methanol, followed by 2-ml of tap water (**NOTE:** Do NOT use distilled water or higher grade water since the aqueous conditioner should be similar to the sample in pH, salt concentration, etc. Use manufacturer specified procedures if they differ from those stated below for conditioning, sample adsorption and elution from the SPE

cartridges.). The tube should not dry up anytime during the conditioning procedure. Disconnect vacuum source before setting up sample extraction.

9.4.3 Using a black marker, mark the meniscus of the water in the sample container.

9.4.4 Add 0.5-mL of the surrogate sample spike solution and mix it thoroughly by gently swirling the bottle.

9.4.5 Add 1-mL of the appropriate spiking solution to LCS/LCSD & MS/MSD samples only.

9.4.6 Dip one end of the sample drain tube into the bottom of the sample bottle and snap the other end into the SPE cartridge. Adjust the flow rate of the sample through the SPE cartridge to less than 10-mL/minute by adjusting the vacuum controller for each sample in the manifold.

9.4.7 After all the samples have passed through the cartridge, but before the SPE tube goes dry, remove the connecting tube from the cartridge and elute the target compounds with 5.0-mL acetonitrile. Pulse the vacuum pump to start the flow, then let the solvent drip through under gravity alone at a flow rate of approximately 1-mL/minute. Collect the acetonitrile extract in clean 10-mL screw-cap tubes.

9.4.8 Collect the extract and perform volume reduction and solvent exchange as described in Section 9.1.4 and 9.1.5. The extract is ready for GC/MS analysis.

9.4.9 Add tap water to the empty sample container up to the marked meniscus. Pour the distilled water into a 1-liter graduated cylinder. Record this measured volume as the sample volume in the logbook. Perform volume measurement for each sample.

10.0 GC/MS CALIBRATION PROCEDURE

10.1 Chromatographic conditions (See also Appendix 1 for documentation):

Injection port: 250°C
Transfer line temperature: 300°C
Split ratio: 5:1
Split flow: 6-ml/min.
Carrier gas flow rate: 15 ml/min.
Initial temperature: 50°C, hold for 2-minutes
Temperature program: 6°C /min
Final temperature: 300°C, hold for 2-minutes

10.2 Mass Spectrometer Conditions for Evaluating GC and MS Performance

Mass range: 40 to 600-AMU
Scan rate: 1-sec/scan
Source temperature: 230°C

10.3 Mass Spectrometer Tune and GC Column Performance - GC column performance and mass spectrometer tune are not critical for the PAHs analysis in the selected ion monitoring mode. However, it is recommended that the GC and MS be tuned in accordance with EPA SW-846 Method 8270C for this analysis.

10.3.1 The gas chromatograph and the mass spectrometer are tuned (see Appendix 1 and 2 for details) to meet the criteria in ***TABLE 4,*** *every 12 hours*, for the injection of 50-ng of DFTPP, 4, 4'-DDT, PCP, & Benzidine tuning solution (1-ul injection of the tuning standard). Inject the tuning standard and using the mass abundances determine if the criteria is met for DFTPP as stated in ***TABLE 4***. Background subtraction should be straight-forward only to eliminate column bleed or instrument background ions. If it is not met, retune the mass spectrometer and reanalyze the DFTPP standard. Although the target analytes for this SOP are not affected very much, it is preferable not to perform initial calibration until the criteria are met.

10.3.2 The GC/MS tuning standard should also be used to assess GC column performance and injection port inertness. DDT breakdown to DDD and DDE should not exceed 20%. Corrective action such as injection port maintenance must be performed when degradation is excessive. The calculation for % breakdown is as follows:

$$\% \text{ breakdown (DDT)} = \frac{\text{Peak area of (DDE + DDD)} * 100}{\text{Peak area of (DDT+DDE +DDD)}}$$

10.3.3 Measure peak tailing (See Appendix 1 for details). Quantitative peak tailing factors of 3.0 and 5.0 are recommended for benzidine and pentachlorophenol respectively. Check also the instrument response for these compounds. Clean the injection port and/or break off the first 10- to 25-cm of the capillary column as needed to restore GC performance. Although the target analytes for this SOP are not affected very much, it is preferable not to perform initial calibration until the criteria are met.

10.4 Initial Calibration (ICAL) - This procedure includes the verification of retention times for the target compounds, surrogates and internal standards and making adjustments to the SIM tables (Table 5) and the ion switching times.

10.4.1 Once the criteria in section 10.3 have passed, analyze a mid-level SVOCs standard (Section 5.20) in the full scan mode to verify retention times of target compounds, internal standards and surrogates (see Table 1). Make adjustments to the SIM table and set up the instrument (see Appendix 1 and 2 for details) to perform initial calibration. Do not change gas chromatographic conditions for the PAHs analysis.

10.4.2 Set up the selected ion monitoring tables and make adjustments to the chromatographic ion switching times (see Table 5) as needed to monitor all the compounds.

10.4.3 Inject the initial calibration solutions prepared as described previously (see Section 5.11.1 or 5.11.2 as appropriate). The calibration curve may be run from high to low instead of low to high. Summarize the initial calibration (See Appendix 3 for details) data in Form-6 equivalents.

10.5Tabulate the area response of the characteristic ions against concentration for each compound and each standard. Document ICAL in Form-6 equivalents (see Appendix 3 for details). Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard that corresponds to the SVOC compounds is shown in ***TABLE 1***. The RF is calculated as shown below:

$$RF = (A_x C_{is}) / (A_{is} C_x)$$

where:
Ax = Area of the characteristic ion for the compound being measured
Ais = Area of the characteristic ion for the specific internal standard
Cis = Concentration of the specific internal standard
Cx = Concentration of the compound being measured

10.6 The average RF must be calculated and recorded for each compound using the five (or more) RF values calculated for each initial calibration solution.

10.7 Using the RFs from the initial calibration for each ICAL standard, calculate and record the percent relative standard deviation (%RSD) for all the compounds. The percent RSD is calculated as follows:

$$\%RSD = ((SD)/(RF_m))*100\%$$

where:
RF_m = Mean of the 5 (or more) RF for the ICAL solutions
SD = standard deviation for each mean RF

10.8 Linearity - If the % RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range and the average relative response factor may be used for quantitation. The analyst, exercising professional judgment, may use regression methods for compounds with less than 15% RSD, if decreasing or increasing trends are observed for response factors at various concentrations. NOTE: The maximum allowable %RSD for any compound is 40% according to Appendix I of the USACE document EM 200-1-3.

10.8.1 If the % RSD of any detected compound is greater then 15%, use least square regression methods to calculate sample concentrations. For regression methods, the instrument response is treated as the dependent variable (y) and the standard amount is the independent variable (x). When "Cs" is used as "x", use "As*Cis/Ais" as "y". When "Cs/Cis" is used as "x", use "As/Ais" as "y". Do not include origin (0,0) as an extra calibration point.

10.8.2 Weighted least squares method is preferred for calculating concentrations more accurately at lower levels. A linear least squares equation (y = a*x + b) is preferred over non-linear calibration equations (y = a*x2 + b*x + c). The coefficient of determination (COD), coefficient of variation (r2) or correlation coefficient (r) should be used to measure the goodness of fit. For regression models to be used for quantitative purposes, the COD, r2 or r must be greater than or equal to 0.99. A value of 1.00 indicates a perfect fit. Corrective action may be required if the criteria for %RSD, r, r2, or COD are not met.

10.8.3 An initial calibration (ICAL) should be considered a single event process. Consult the QA office or check applicable EPA methods (e.g. SW-846 Method 8000C and 8270D) for additional guidance on appropriate corrective action.

10.8.4 Re-fitting of ICAL – The selected model for ICAL may be subjected to an additional re-fitting check to establish representativeness of the model for each compound in each ICAL standard. This is an optional procedure, which is recommended but not required since automated data processing procedures may not be able to accommodate it. Calculate the %D between the calculated amount (Cc) for each compound in each ICAL standard from the ICAL model and its expected value (Ce). Ideally, the %D ((Cc-Ce) * 100 /Ce) for each compound in ICAL standard should meet the QC criteria for %RSD. It is possible for one or more ICAL standards to have %D exceeding the %RSD criteria, while the %RSD for ICAL model is within QC limits. The re-fitting procedure may be used to identify ICAL standards that need re-analysis or dropped from the calculation of ICAL model when re-analysis was not performed.

10.8.5 Check and optimize instrument-operating conditions including automated peak integration procedures for appropriateness in addressing the chromatographic needs of the specific compound with problems.

10.8.6 If the problem appears to be associated with just one of the ICAL standards, that one standard may be reanalyzed once immediately (must be within 8-hours) after the ICAL. Discard all the results from the original analysis of the standard in question and replace it with the results from re-analysis.

10.8.7 When additional calibration standards (six or seven instead of five) are analyzed, narrowing the range of concentrations to be used for determining the goodness of fit is an acceptable procedure. Five or more standards should remain for the calculation of RSD or linear regression for each compound after one or more points are removed from the ICAL. More points (6 for second order and 7 for third order) may be needed for non-linear regression. It is generally prohibited to remove data points from within a calibration range, while still retaining the extreme ends of the calibration range. Do not discard a middle point unless it appears to have an obvious problem and six or more standards were analyzed for ICAL and re-analysis of the affected standard was not performed within the required time limits. Changes to the upper end of the calibration range will affect the need to dilute samples above the range. A change to the lower range is not recommended since it will affect the method quantitation limit (MQL) used to report data. The default MQLs, which are based on the lowest ICAL concentration are stored in the LIMS and project-specific changes are not practical.

10.8.8 The refitting procedures may lead to failed %Ds for upper level calibration standards for average response factor methods. The analyst may take alternate corrective actions such as the analysis of sample extracts under dilution for the affected compounds.

10.8.9 The refitting procedures may lead to failed %Ds for lower level calibration standards for regression methods. The analyst may take appropriate corrective action such as flagging the sample result as an estimated amount ("J" flag) at the lower level encountered in the sample.

10.8.10 **NOTE: Grand mean RSD** – Previous versions of the EPA method(s) had provisions for the calculation of the grand mean %RSD for all target

compounds in the ICAL solution, when the % RSD of any detected compound is greater than 15%. The grand mean %RSD should be <15% for ICAL to be acceptable. This procedure is discontinued. Criteria for Initial calibration are compound-specific.

10.9 Tuning verification - Since tuning verification for every 12-hour shift will involve reconfiguration of the instrument, instrument tune need not be checked every 12-hours. Verify response factors every 12 hours.

10.10 Calibration verification (CALV) – It consists of two steps: an initial calibration verification (ICV) and continuing calibration verification (CCV);

10.10.1 The initial calibration verification (ICV) standard is prepared using a second source PAH standard. Analyze a mid-level second source standard (see Section 5.18.3) immediately after the analysis of ICAL solutions.

10.10.2 Continuing calibration verification (CCV) is performed using the mid-level standard (see Section 5.19) that is identical to the mid-level standard used to generate the ICAL curve.

10.10.3 A CCV is not necessary for samples analyzed immediately after ICAL and ICV provided they are within 12-hours of the mid-level ICAL standard.

10.10.4 Use standard SW-846 Method 8270c/8270D procedures for ICVs and CCVs.

10.10.5 Summarize ICV and CCV in Form-7 equivalents (See also Appendix 4 for details).

10.10.6 Calculate either the percent difference (%D) (for response factors) or percent drift (%D) (for linear regression) for the CCCs using one of the following equations:

$$\%D = \%Difference = ((RF_v - RF_m) * 100)/(RF_m)$$

$$\%D = \%\ drift = ((C_v - C_m) * 100) / (C_m)$$

where:

RF_m = Average or mean response factor from ICAL
RF_v = Observed RF for the CALV standard
C_m = True concentration of the CALV standard.
C_v = Measured concentration of the CALV standard using selected quantitation method (e.g. linear regression).

10.11 For specific USACE projects, client may specify that %D for the contaminants of concern (COC) should be within the CCV QC limits (<20%).

10.12 For USACE projects, the %D for all target analytes should be <30% for initial calibration verification (ICV). This procedure employs a second source standard for the verification, which is uncommon for most organic EPA methods.

10.13 **NOTE: Grand mean %D** – Previous versions of the EPA method(s) had provisions for the calculation of the grand mean %D for all target compounds in the CALV solution, when the %D of any detected compound is greater than 20%. The grand

mean %D should be <20% for CALV to be acceptable. ***This procedure is discontinued.*** Criteria for calibration verification are compound-specific.

10.14 The internal standard responses and retention times in the check calibration standard must be evaluated immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration check (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made, as required.

10.15 If the EICP area for any of the internal standards (in all analyses) changes by a factor of two (-50% or + 100%) from the last daily calibration check, the mass spectrometer must be inspected for malfunctions and corrections must be made as necessary. If any samples were analyzed after the system checks are invalid, those samples will need to be re-analyzed after corrections have been made.

11.0 ANALYSIS OF EXTRACTED SAMPLES

11.1 Once the samples have been extracted and all system performance checks have been established as stated in the previous sections, sample extracts can be analyzed. Each 1-mL aliquot is spiked with 10-uL of the appropriate internal standard solution (see Section 5.10.1 or 5.10.2). The sample extracts and associated quality control sample (MB, LCS, LCSD, MS, and MSD) extracts are analyzed in the same manner as calibration standards. Document analysis on instrument logs (See Appendix 13 for details).

11.2 If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, reanalyze sample extracts after appropriate dilutions. Additional internal standard must be added to the diluted extract to maintain the required level for each internal standard in the extract that is injected. The diluted extract must be reanalyzed.

12.0 DATA INTERPRETATION

12. 1 The qualitative identification of compounds determined by this method is based on retention time and the presence of the quantitation (or primary) and secondary ions at the RT window established.

12.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

12.1.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

12.1.3 Identification can become difficult when more than one analyte elutes chromatographically causing overlapping of spectra.

13.0 QUANTITATIVE ANALYSIS

13.1 The internal standard technique is used to determine the concentration of an identified target compound. For water samples the calculation is as follows:

$$\text{Concentration } (\mu g/L) = \frac{(A_X) * (IS) * V_e}{(A_{IS})\,(RF_m)\,(V_s)}$$

where:

A_X = Area of characteristic ion for compound being measured.
IS = Amount of internal standard injected (µg/mL)
A_{IS} = Area of characteristic ion for the internal standard
RF_m = Mean RF for compound being measured from the ICAL
V_e = Volume (mL) of the final sample extract, taking into account any dilutions made.
V_s = Volume (Liter) of water extracted

For soil samples the calculation is as follows:

$$\text{Concentration } (\mu g/kg) = \frac{(A_X)\,(IS)\,(V_e)}{(A_{IS})\,(RF_m)\,(W_S)\,(PS/100)}$$

where:

A_X = Area of characteristic ion for compound being measured.
IS = Amount of internal standard injected (µg/mL)
A_{IS} = Area of characteristic ion for the internal standard
RF_m = Mean RF for compound being measured from the ICAL
V_e = Volume of total extract (mL)
W_S = Weight of sample (kg) extracted
PS = Percent Solids

13.2 Document sample data in quantitation reports and sample chromatograms (see Appendix 5 for details). Report sample results in Form-1 equivalents (see Appendix 6 for details).

14.0 QUALITY CONTROL

Analytical data, which includes calibration data, sample preparation logs, sample data and quality control data should be gathered prior to review. Review checklist (See Appendix 7 for details) should be used to document verification of calculations and adherence to method procedures. Exceptions should be documented. Samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions should be documented on these checklists.

14.1 Summarize sample results using sample result Form 1 equivalents (See Appendix 6 for details). Use the same format for all analyses including QC samples such as MB, LCS, LCSD, MS, and MSD. Evaluate acceptability of analysis results using Form-8 internal standard area and retention time summary (See Appendix 9 for details). The MDLs, RLs (when applicable), and MQLs applicable to each matrix and/or method are shown in Tables 2 and 3. Report positive results greater than the MDL and/or the RL but

less than the MQL as estimated with a "J" flag. Report positive results that are greater than the upper calibration limit as estimated with an "E" flag. Evaluate sample data as shown in the Data Verification section and apply additional qualifiers as needed. Document them in the checklists (see Appendix 7 for details) and in the case narrative provided to clients.

14.2 Summarize QC results for LCS/LCSD in Form-1 and recoveries in Form-3 equivalents (see Appendix 10 for details). Evaluate them as shown in the Data Verification section.

14.3 Summarize QC results for MS/MSD in Form-1 and recoveries in Form-3 equivalents (see Appendix 11 for details). Evaluate them as shown in the Data Verification section.

14.4 Summarize surrogate recoveries in Form-2 equivalents (see Appendix 12 for details). Evaluate them as shown in the Data Verification section.

14.5 Summarize method blank (MB) results in the same manner as sample results (see Appendix 6 for details). Summarize MB or QC association in Form-4 equivalents (see Appendix 13 for details). Evaluate them as shown in the Data Verification section. Report target compounds that are present in the MB at levels greater than the reporting limit (RL) using J qualifiers if the levels are less than PQL.

15 DATA VERIFICATION AND VALIDATION - This section will provide guidance when evaluating and validating data generated by this SOP. It provides procedures to assess if data produced is valid and legally defensible. Document review and comments on the checklist (See Appendix 7). Verify that sample preparation logs are complete and the instrument run logs are available for review. Anomalies and exceptions should be documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on these checklists. USACE projects may have special requirements that will require extra attention. A simple, universal procedure for evaluating choosing appropriate corrective action doe not exist to deal with situations when one or more QC parameters are outside the acceptance window. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints.

15.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) projects may have additional requirements that are related to USACE laboratory validation (CX), USACE District requirements, government contract requirements, specific USACE project requirements or USACE Architect/Engineer (A/E) requirements. USACE-CX requirements (Summary of Method Quality Objectives for Method 8270C) are included in Appendix 8.

15.1.1 The USACE requirements (see Appendix 8 for details) and elsewhere in this SOP are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may be unattainable for routine sample analysis. Since USACE MQOs are simply

objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. The laboratory may employ default QC limits based on a combination of DoD QSM limits, USACE Shell limits, laboratory statistical limits, or other administrative limits. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

15.1.2 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality objectives and/or other QC requirements. Exceptions and data qualifications necessary should be documented on the checklists and eventually on case narratives.

15.1.3 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist (see Appendix 7 for details), perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete. For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete. For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director will review the checklists (initial/date as necessary), prepare case narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

15.1.4 The blank criteria for USACE projects (see Appendix 8 for details) has been modified by USACE auditor(s) as one half of the applicable PQL (personal communications, USACE auditor, 1999).

15.2 HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

15.2.1 WATER - The holding times for semi-volatiles is 7 days from sampling.

15.2.2 SOIL – A 14 day holding is used for soil and waste samples.

15.2.3 TCLP leaching should be performed within 14 days of waste sample collection. TCLP leachates (aqueous samples) must be extracted within 7 days of leachate preparation.

15.2.4 All sample extracts should be analyzed within 40 days after extraction.

15.2.5 Holding times are established by comparing the chain of custody sampling date with the date of the injection after purging. TCLP holding times must be established by comparing first the COC to the leaching date and then the leaching date to the injection date.

15.2.6 ACTION – If holding times have been exceeded then all positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or invalid (I).

15.3 INSTRUMENT TUNING/PERFORMANCE – Tuning and performance criteria for mass resolution, identification and sensitivity should meet criteria established in ***TABLE 4***. DDT breakdown to DDD and DDE should not exceed 20%. Peak tailing factors for benzidine and pentachlorophenol should be below 3.0 and 5.0 respectively. Perform appropriate corrective action as described in this SOP to restore chromatographic performance to acceptable levels. NOTE: The instrument performance criteria described in this section and the referenced SW-846 method are not strictly applicable since the instrument is not operating in the full scan mode and target compounds do not include phenols and breakdown pesticides such as DDT. However, they should be used as criteria to assess the condition of the instrument and to take appropriate corrective action.

15.3.1 Compare mass calibration from tabular report in the raw data with each criteria form.

15.3.2 Check to see if the tuning report is present for each 12-hour period of samples analyzed.

15.3.3 Verify that the spectrum is present.

15.3.4 Calculate % abundance.

15.3.5 ACTIONS – If mass calibration is outside criteria, recalibrate and rerun tuning standard for an acceptable tune report. Reanalyze all associated samples from failed tune. If mass calibration is outside criteria, all associated data may be qualified as estimated (J) or invalid (I) by the client at client's discretion.

15.4 INITIAL CALIBRATION – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the mass spectrometer is capable of producing data within a range that is linear. See also additional client-specific initial calibration requirements including second-source ICV analysis (see Appendix 8 for details).

15.4.1 Target compounds – Check to see if the %RSD for any of the target compounds is outside the 15% criteria or less for %RSD. If a compound is outside the 15% criteria, check to see if linear regression can be used. The lower limit for linear regression is 0.99.

15.4.2 ACTION – Positive results for compounds not meeting the criteria may need to be qualified as estimated (J) by the user (client). Report detects and non-detects without qualifiers and mention these outliners in the checklist and in the case narrative.

15.5 CALIBRATION VERIFICATION – This includes ICV and CCV. The ICV is used to check the reliability of the primary standard used for ICAL. The continuing calibration verification (CCV) is used to check that the instrument is still operating under the linear range of the initial calibration and has remained stable every 12 hours. See also additional client-specific calibration verification requirements (Appendix 8).

15.5.1 Target compounds – Check to see if any of the target compounds have %D outside the 20% criteria.

15.5.2 ACTION – Positive results for samples not meeting the criteria for the CCC and SPCC criteria may be qualified as estimated (J) or invalid (I) by the user (client) at their discretion. Any positive results for compounds other than CCC and SPCC not meeting the criteria may be qualified as estimated (J) by the user (client). Report data without qualifications documenting the QC outliers in the checklist and in the case narrative.

15.6 METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. The processing of blanks helps eliminate reporting of false positives.

15.6.1 Method blanks should be free of all target analytes and other interfering non-target analytes. To be acceptable, blank results should not exceed one half of the PQL for any compounds with the exception of common plasticizers such as phthalate esters.

15.6.2 Review the results of all associated blanks and verify that the method blank has been reported per matrix for each system used to analyze the associated samples. If levels greater than one half the MQLs are present, inspect and correct the problem. Reanalyze all associated samples. The only exception may be quick turn around projects.

15.6.3 ACTION – All positive results are reported with a “B” code if contaminants are found in the method blank.

15.6.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, reanalysis of the samples is not required.

15.6.5 ACTION – Any compound detected in the sample which were also detected in associated method blank both being greater than one half of the MQLs (except phthalate esters) should be re-extracted.

15.6.6 ACTION – If contaminants resulting in GC/MS peak saturation are present, all affected compounds are qualified as invalid (I).

15.7 SURROGATE RECOVERIES – Sample quality control is measured by means of spiking individual samples with system monitoring compounds which are different structurally, but are chemically similar to classes of target compounds. Three base neutral (BN) and three acid (A) surrogates are spiked into the samples just prior to when the extracting solvent is added to the sample. Only the BN surrogates are applicable to the PAHs analysis. Due to the limited number of target compounds three BN surrogates need not be used for the PAHs analysis. One surrogate may be used at the discretion of

the analyst. When two or more (three) surrogates are used, two of the surrogates should be within the QC limits for sample analysis to be acceptable.

15.7.1 Verify surrogate recoveries by comparing recovery limits shown in ***TABLE 6*** with the observed recoveries in the samples.

15.7.2 ACTION – When one or more of the BN surrogate recoveries are outside QC limits, the sample must be re-analyzed. Reanalysis may be performed under dilution if original analysis should indicate high levels of target and/or non-target analytes.

15.7.3 ACTION – When any of the surrogate recoveries are outside the QC limits for controlled matrices such as MB, LCS and LCSD, it may indicate problems with method performance. Problems should be identified and corrected as soon as possible, preferably prior to the analysis of additional samples. QC criteria described above also applies to these samples.

15.8 LABORATORY CONTROL STANDARD (LCS) – A LCS analysis is used to evaluate method competence in the absence of matrix effects. Since a variety of matrices are encountered during sample processing. The LCS may provide a mechanism to check method efficiency for target compounds without the sample matrix as a factor. The LCS is evaluated by comparing the percent recovery for the entire target analytes to the laboratory QC limits, which are identical to the 3-sigma DoD QSM QC limits. See Appendix 10 for guidance on documenting LCS and LCSD concentrations and %R in QC summary report forms. The DoD QSM specifies the number of compounds that can exceed the 3-sigma QC limits based on statistical considerations. The LCS recovery for such compounds should not exceed the marginal exceedance 4-sigma (ME) limits. Evaluation procedures may include comparison of LCS recoveries with those determined by project-specific DQOs (See project QAPP, if available), laboratory statistical control limits, or USEPA guidance. Initially, the effect of QC failure on the samples should be evaluated. Regardless of this assessment, steps shall be taken to find the source of the problem and correct it.

15.8.1 CHECK - The DoD QSM control and ME limits for LCS/LCSD recoveries are shown in Tables 2 and 3. Compare the %R results for each compound in LCS with laboratory QC limits to see if all the results are within the acceptable range. Flag outliers with "ME" if it is within the 3-sigma limits. Flag it with "ME*" if it is outside the 4-sigma ME limits.

15.8.2 CHECK - Compare observed %R results for each compound with the laboratory limits for LCS/LCSD recoveries (e.g. USACE criteria). Perform manual calculation for %R, if necessary for QC outliers. Document the number of compounds that exceeded the QC limit and compare it against the maximum recommended by DoD QSM. Document this review in checklists (Appendix 7) and in case narratives.

15.8.3 ACTION - Typically, the LCS would be reanalyzed for the failed analytes (ME*) only. If the second analysis fails, then the LCS, method blank, and all associated samples of the batch would be reanalyzed for the failed analytes only. If sufficient sample is not available for reanalysis, or if the corrective action is ineffective, discuss the situation with the client project manager (or

chemist). Include a discussion of the situation in the case narrative. The case narrative should discuss the corrective action taken and any other information.

15.8.4 ACTION - For non-USACE projects, no action may need to be taken on LCS and/or LCSD recovery data alone either to reanalyze samples and/or to qualify associated samples. Providing data of known quality is sufficient for most projects. If significant deviations (e.g. number of target compound exceeding QC limits for recoveries is greater than the allowable maximum) are noticed, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements, which may be compound specific. Check with the project manager about such requirements. Professional judgment may be used by the user (client) in evaluating sample data qualification options in conjunction with other QC data for the project.

15.9 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike data are used to evaluate the precision and accuracy of the analytical method on project samples. See also client-specific guidelines for MS (see Appendix 8 for details).

15.9.1 MS data evaluation is more complex than method blank or LCS data evaluation since they are complicated by matrix effects in addition to sample preparation and analysis errors. The heterogeneity of grab soil samples, and potential non-representativeness of sequentially collected water samples, etc. further complicate the evaluation since it is assumed that the native concentrations are constant in triplicate analyses. In addition, concentrations of the target analytes in the sample can also far exceed spike amounts. If the native concentration of target analytes in the sample chosen for spiking is high relative to the spiking concentration, the differences in the native concentration between the unspiked sample and the spiked samples may also become very significant.

15.9.2 In addition to their reluctance to pay for MS and/or MSD analyses, most clients may not know what samples to collect for MS analysis. Clients may not collect sufficient samples, have no knowledge of the native analyte concentrations in the chosen sample, and may not have knowledge of MS spiking levels that are appropriate either for the sample or their project. For procedures with multiple target analytes, spiking levels may be inappropriate for one or more analytes, if samples that contain target analytes are chosen either in the field or by the laboratory. If no project samples are collected for MS analysis, samples from another project used for MS analysis may not be representative of the project samples. When project samples (e.g. surface soil) are chosen, their MS recovery characteristics may be different from those of other samples (e.g. sub-surface soil) in the same batch.

15.9.3 MS/MSD recovery limits are shown in Tables 2 and 3. The relative percent difference (RPD) between the duplicate analyses (MS and MSD) are also included in these tables. Document MS/MSD recoveries in instrument printouts and in LIMS generated reports as shown in Appendix 11. Flag QC outliers with “ME” and “ME*” on client reports.

15.9.4 CHECK - Compare %R results for each compound with the laboratory limits for MS/MSD recoveries as described for LCS. Perform manual calculation for %R using raw data and extraction sheet information to check outliers. Document this review in checklists (Appendix 7) and in case narratives.

15.9.5 ACTION – The results of the MS are evaluated, in conjunction with other QC information, to determine the effect of the matrix on the bias of the analysis. For DoD/USACE projects, acceptance criteria may allow for marginal exceedances as described for LCS (See above). Although these criteria are not applicable to LCS data, steps should be taken to find the cause of failure and corrective action to remedy it. Matrix spike recoveries that fail to meet the appropriate acceptance criteria may indicate that a potential matrix effect is present. A review of the MSD result, if available, may confirm the matrix effect, if it is in the same direction and same order of magnitude. Check the LCS and/or LCSD recoveries for affected compounds to rule out laboratory problems.

15.9.6 ACTION - If the sample chosen for MS/MSD is inappropriate, notify the client. When additional payments are authorized for re-analysis, re-extract and/or reanalyze the MS/MSD samples after employing cleanup procedures (if applicable), dilution techniques to minimize matrix interference, or using other project samples that appear to be clean, when sufficient quantities are available. Re-spike the sample at a higher level (e.g., at approximately two to four times the sample concentration), then reanalyze the sample based on project-specific requirements. It is the responsibility of the client at all times to select appropriate samples for MS analysis, make payments for additional analyses, and to offer necessary guidance as needed to meet their data quality objectives.

15.9.7 ACTION - If the matrix effect cannot be resolved, discuss the situation in the case narrative. No action may be taken by the laboratory to qualify associated sample data based on MS/MSD recovery data alone. Professional judgment may be used by the user (client) in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project. Document QC outliers on the checklist, client reports (*) and draw attention to the QC outliers on the case narrative.

15.10 INTERNAL STANDARD PERFORMANCE – Document internal Standard (IS) areas and retention times in Form-8 equivalents (See Appendix 9 for details). Adhering to the criteria ensures that the GC/MS sensitivity and response are stable during each run. Six internal standards are not necessary for quantitating the 16 PAH compounds. The analyst may use a lesser number for quantitation. The same internal standards should be used for calibration (ICAL, ICV, and CCV) and sample analysis.

15.10.1 IS area counts should not vary by more than a factor of 2 (-50% or + 100%) from the associated ICAL/Calibration Verification standard. The internal standard areas for the CCV should not differ by a factor of 2 (-50% or + 100%) from the initial calibration of the same standard.

15.10.2 The retention time of the internal standard should not vary by more than 30 seconds from the associated daily calibration standard.

15.10.3 ACTION – Document IS areas and RT on Form-8 equivalents (see Appendix 9 for details). If an internal standard is outside of QC limits of the associated daily standard, then re-analysis is required. Reanalysis under dilution may be required if it appears that interference's occur from target and/or non-target analytes at high concentrations relative to the ICAL. Do not dilute the sample to the point where all target analytes are non-detect. Professional judgement must be used when dilutions are required for IS criteria.

15.10.4 ACTION – If an internal standard is outside of QC limits of the associated ICAL or CCV standard, re-analyze the affected sample(s) first. If re-analysis has been performed and it is still out, it is of the professional judgement of the analyst to dilute or not to dilute the sample further so as to not to dilute out any target analytes. Report the results for the most acceptable (or report both analyses depending on project requirements) analysis and document the problem on the checklist and on the case narrative. The user (client) may qualify positive results for compounds quantitated using the affected IS as estimated (J). If a very low IS area is reported, indicating a loss of sensitivity, non-detects may be qualified as invalid (I) by the user. Document problems and recommendations in the case narrative.

15.10.5 ACTION – If an IS retention time varies by more than +/- 30 seconds, the total ion profile must be checked for any false positives or negatives. If a large shift has occurred, then professional judgement must be used as to whether reanalyze or not.

15.11 TARGET COMPOUND IDENTIFICATION – Criteria for GC/MS qualitative analysis (Section 12.1) have been provided to minimize the number of erroneous positive or negative identifications of compounds.

15.11.1 For positive identification of the target compound, its RT must be within +/- 0.06 relative time (RRT) units of the standard RRT.

15.11.2 ACTION – Professional judgement must be used when applying the criteria for GC/MS analysis of target analytes. The extracted ion profiles should match fairly well for the sample and standard, taking into account factors such as matrix ions and column bleed.

16.0 REFERENCES

16.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), December 1996.

16.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 4, January 1998.

16.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.

16.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

16.5 Instrumental Analysis and Quantitation of Polycyclic Aromatic Hydrocarbons and Atrazine: IADN Project, Version 1.3, July 1996, D. Cortes, and W. Brubaker, School of Public and Environmental Affairs, Indiana University, Bloomington, IN 47405.

16.6 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3500B, Organic Extraction and Sample Preparation, December 1996.

16.7 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3500C, Organic Extraction and Sample Preparation, Update IV-B.

16.8 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3520C, Continuous Liquid-Liquid Extraction, December 1996.

16.9 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3510C, Separatory Funnel Liquid-Liquid Extraction, December 1996.

16.10 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3535, Solid Phase Extraction, December 1996.

16.11 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3535A, Solid-Phase Extraction, Update IV-B.

16.12 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3540C, Soxhlet Extraction, December 1996.

16.13 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3541, Automated Soxhlet Extraction, December 1996.

16.14 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3545, Pressurized Fluid Extraction, December 1996.

16.15 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3550B, Ultrasonic Extraction, December 1996.

16.16 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3600C, Cleanup, December 1996.

16.17 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3640A, Gel-Permeation Cleanup, December 1996.

16.18 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3580A, Waste Dilution, December 1996.

16.19 Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, EPA, 40 CFR Part 136, Method 625: Base Neutral and Acid Compounds, October 1984.

16.20 Methods for the Determination of Organic Compounds in Drinking Water, Supplement II, EPA/600/4-88-039, Method 525.1, Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry, July 1991.

16.21 AML Standard Operating Procedures, O-BNA-SVOCs-RB.

16.22 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.

16.23 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

16.24 AML Standard Operating Procedures, Q-Water-RC.

16.25 AML Standard Operating Procedures, Q-WasteMgmt-RD.

16.26 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

16.27 AML Standard Operating Procedures, Q-Controlcharts-RC.

TABLE 1
CHROMATOGRAPHIC RETENTION TIMES (DB-5 COLUMN) AND SELECTED IONS FOR MONITORING BY GC/MS FOR POLYNUCLEAR AROMATIC HYDROCARBON COMPOUNDS

ANALYTE	RETENTION TIME (minutes)	INT STD	Primary (m/z)	Secondary (m/z)
TARGET COMPOUNDS				
TC-1 Naphthalene	8.54	IS-2	128	-
TC-2 Acenaphthylene	14.18	IS-3	152	151
TC-3 Acenaphthene	14.91	IS-3	154	153
TC-4 Fluorene	16.80	IS-3	166	165
TC-5 Phenanthrene	20.21	IS-4	178	176
TC-6 Anthracene	20.38	IS-4	178	176
TC-7 Fluoranthene	24.63	IS-4	202	101
TC-8 Pyrene	25.36	IS-5	202	101
TC-9 Benzo(a)anthracene	29.99	IS-5	228	114
TC-10 Chrysene	30.11	IS-5	228	114
TC-11 Benzo(b)fluoranthene	33.77	IS-6	252	126
TC-12 Benzo(k)fluoranthene	33.86	IS-6	252	126
TC-13 Benzo(a)pyrene	34.73	IS-6	252	126
TC-14 Indeneo(1,2,3-cd)pyrene	38.04	IS-5	276	138
TC-15 Dibenz(a,h)anthracene	38.24	IS-6	278	139
TC-16 Benzo(g,h,i)perylene	38.66	IS-6	276	138
INTERNAL STANDARDS				
IS-1. 1,4-Dichlorobenzene-d4	4.72	Not Used	152	-
IS-2. Naphthalene-d8	8.49		136	-
IS-3. Acenaphthalene-d10	14.80		164	-
IS-4. Phenanthrene-d10	20.14		188	-
IS-5. Chrysene-d12	30.03		240	-
IS-6. Perylene-d12	34.93		264	-
SURROGATE COMPOUNDS				
AS-1 2-Flouorophenol	NA	Not Used		
AS-2 Phenol-d5	NA	Not Used		
BNS-1 Nitrobenzene-d5	6.36	IS-2	82	-
BNS-2 2-fluorobiphenyl	12.67	IS-3	172	-
AS-3 2,4,6-tribromophenol	NA	Not Used		
BNS-3 Terphenyl-d14	26.62	IS-5	244	-

NOTE: Base neutral (BN) surrogates are used. The acid surrogates (A), although spiked into some samples are not used for recovery determination.

TABLE 2
Aqueous MDLs, MQLs, and QC Limits for LCS and MS

Polynuclear Aromatic Hydrocarbons (PAHs) Analysis by Selected Ion Monitoring (SIM) GC/MS

Analytical Management Laboratories

Matrix: Water	Preparation Method			3510C										
	Analytical Method			8270D-SIM										

	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**			**DoD QSM Control Limits LCS & MS**		**DoD QSM ME Limits LCS & MS**		**RPD**
Compound	Amount	ug/L	ug/L	**ug/L**	RD=B/A	flag	**ug/L**	RQ=C/A	flag	Lower	Upper	Lower	Upper	Limit	
	ug/L	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%	
Naphthalene	0.05	0.047	0.013	**0.035**	1.3		0.2	5.7		40	100	30	115	50	
Acenaphthene	0.05	0.033	0.007	**0.019**	1.7		0.2	10.5		45	110	35	120	50	
Acenaphthylene	0.05	0.031	0.006	**0.016**	1.9		0.2	12.5		50	105	40	115	50	
Fluorene	0.05	0.042	0.008	**0.022**	1.9		0.2	9.1		50	110	40	120	50	
Anthracene	0.05	0.133	0.005	**0.014**	9.5	*	0.2	14.3		55	110	45	120	50	
Phenanthrene	0.05	0.060	0.009	**0.027**	2.2		0.2	7.4		50	115	40	130	50	
Fluoranthene	0.05	0.144	0.005	**0.015**	9.6	*	0.2	13.3		55	115	45	125	50	
Benzo(a)anthracene	0.05	0.070	0.015	**0.042**	1.7		0.2	4.8		55	110	45	120	50	
Chrysene	0.05	0.035	0.007	**0.020**	1.8		0.2	10.0		55	110	45	120	50	
Pyrene	0.05	0.034	0.005	**0.015**	2.3		0.2	13.3		50	130	35	140	50	
Indeno(1,2,3-cd)pyrene	0.05	0.067	0.021	**0.058**	1.2		0.2	3.4		45	125	30	140	50	
Benzo(b)fluoranthene	0.05	0.133	0.005	**0.014**	9.5	*	0.2	14.3		45	120	35	130	50	
Benzo(k)fluoranthene	0.05	0.099	0.003	**0.009**	11.0	*	0.2	22.2		45	125	30	135	50	
Benzo(g,h,i)perylene	0.05	0.035	0.008	**0.024**	1.5		0.2	8.3		40	125	25	135	50	
Benzo(a)pyrene	0.05	0.039	0.007	**0.021**	1.9		0.2	9.5		55	110	45	120	50	
Dibenz(a,h)anthracene	0.05	0.030	0.007	**0.019**	1.6		0.2	10.5		40	125	30	140	50	
Mean Ratio (RD or RQ):					3.8			10.6							

Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 5 and MQL/MDL ratios (RQ) that are less than 3.

MDL data from two spike levels were combined for this table. QC limits for RPDs are from EM 200-1-3, Appendix I (Feb 2001).

The 8270-C LCS and MS %R control limits and marginal exceedance (ME) limits are from the DoD QSM, Revision 2 Final, June 2002.

Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).

NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.

TABLE 3
Soil/sediment MDLs, MQLs, and QC Limits for LCS and MS

Polynuclear Aromatic Hydrocarbons (PAHs) Analysis by Selected Ion Monitoring (SIM) GC/MS														
Analytical Management Laboratories														
Matrix: Soil	Preparation Method			3550C										
	Analytical Method			8270D-SIM										
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**			**DoD QSM Control Limits LCS & MS**		**DoD QSM ME Limits LCS & MS**		**RPD**
Compound	Amount	Ug/kg	ug/kg	**ug/kg**	RD=B/A	flag	**ug/kg**	RQ=C/A	flag	Lower	Upper	Lower	Upper	Limit	
	ug/kg	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%	
Naphthalene	3.33	2.19	0.226	**0.68**	3.2		6.67	9.8		40	105	30	120	60	
Acenaphthene	3.33	2.09	0.129	**0.39**	5.4		6.67	17.2		45	105	35	115	60	
Acenaphthylene	3.33	2.65	0.143	**0.43**	6.2		6.67	15.6		45	105	35	115	60	
Fluorene	3.33	2.35	0.195	**0.59**	4.0		6.67	11.4		50	110	40	115	60	
Anthracene	3.33	2.18	0.262	**0.79**	2.8		6.67	8.5		55	105	45	115	60	
Phenanthrene	3.33	2.28	0.275	**0.83**	2.8		6.67	8.1		50	110	40	120	60	
Fluoranthene	3.33	2.39	0.362	**1.09**	2.2		6.67	6.1		55	115	45	125	60	
Benzo(a)anthracene	3.33	2.78	0.436	**1.31**	2.1		6.67	5.1		50	110	40	120	60	
Chrysene	3.33	2.32	0.306	**0.92**	2.5		6.67	7.3		55	110	45	120	60	
Pyrene	3.33	2.14	0.218	**0.65**	3.3		6.67	10.2		45	125	35	135	60	
Indeno(1,2,3-cd)pyrene	3.33	2.45	0.520	**1.56**	1.6		6.67	4.3		40	120	25	135	60	
Benzo(b)fluoranthene	3.33	2.21	0.374	**1.12**	2.0		6.67	5.9		45	115	35	125	60	
Benzo(k)fluoranthene	3.33	2.39	0.422	**1.26**	1.9		6.67	5.3		45	125	30	135	60	
Benzo(g,h,i)perylene	3.33	2.19	0.335	**1.00**	2.2		6.67	6.6		40	125	25	140	60	
Benzo(a)pyrene	3.33	2.69	0.374	**1.12**	2.4		6.67	5.9		50	110	40	120	60	
Dibenz(a,h)anthracene	3.33	2.19	0.200	**0.60**	3.6		6.67	11.1		40	125	25	140	60	
Mean Ratio (RD or RQ):								8.7							

Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 10 and MQL/MDL ratios (RQ) that are less than 3.

MDL studies were performed at two spike levels and the data combined as shown above. QC limits for RPDs are from EM 200-1-3.

The 8270-C LCS and MS %R control limits and marginal exceedance (ME) limits are from the DoD QSM, Revision 2 Final, June 2002.

Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).

NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.

TABLE 4
DFTPP MASS INTENSITY SPECIFICATIONS

MASS	INTENSITY REQUIRED (RELATIVE ABUNDANCE)
51	30-60% of mass 198
68	<2% or mass 69
70	<2% or mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9 5 mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

TABLE 5
SELECTED IONS FOR MONITORING AND CHROMATOGRAPHIC SWITCHING TIMES

Retention Time (minutes)		Ions monitored (m/z)	Compounds Monitored (See Table 1 for Compound ID)
From	**To**		
0.0	10.0	68, 82, 128,136, 150, and 152	TC-1, BNS-1, IS-1, and IS-2
10.0	18.50	151, 152, 153, 154, 164, 165, 165, and 172	TC-2, TC-3, TC-4, BNS-2, and IS-3
18.50	28.50	94, 101, 122, 176, 178, 188, 202, and 244	TC-5, TC-6, TC-7, TC-8, BNS-3, and IS-4
28.50	36.50	1114, 120, 126, 228, 240, 252, 260, and 264	TC-9, TC-10, TC-11, TC-12, TC-13, IS-5 and IS-6
36.50	42.0	138, 139, 276, and 278	TC-14, TC-15, and TC-16

NOTE: See Table 1 for compound identification (TC-1, BNS-1, IS-1, etc.), ions monitored for each compound, and their chromatographic retention times.

TABLE 6
SURROGATE SPIKE RECOVERY LIMITS
(from DoD QSM, Final Version 2, June 2002)

Surrogate Compound	Percent Recovery			
	Water		Soil / Sediment	
	Lower	Upper	Lower	Upper
Nitrobenzene-d5	40	110	35	100
2-fluorobiphenyl	50	110	45	105
Terphenyl-d14	50	135	30	125

Table 7
LIST OF APPENDICES

Appendix Number	Appendix Topic
1	Chromatographic Conditions for Instrument Evaluation DFTPP Tune Summary (Form-5 equivalents) Instrument Printouts
2	Chromatographic Conditions for PAHs Analysis in the SIM mode Instrument Printouts
3	Initial Calibration (ICAL) Summary (Form-6 equivalents)
4	Calibration Verification (ICV and CCV) Summary (Form-7 equivalents)
5	Quantitation Reports and Chromatograms Instrument Printouts
6	Sample Reports (Form-1 equivalents) - Soil and Water
7	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
8	Method Quality Objectives (MQOs) for Method 8270C (USACE, AFCEE, DoD MQOs, etc.)
9	Internal Standard Area and RT Reports (Form-8 equivalents)
10	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
11	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
12	Surrogate Recovery Summaries (Form-2 equivalents)
13	QC Association Forms (Form-4 equivalents) Sample Preparation Log Instrument Analysis Log

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. O-SoilVOCs-5035A-Text-RC

SOIL SAMPLE COLLECTION AND PRESERVATION PROCEDURES FOR THE ANALYSIS OF VOCs BY SW-846 DRAFT METHOD 5035A

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) is based on SW-846 Method 5035 and Draft Method 5035A for the collection of soil, sediment, and waste samples to be analyzed by closed-system purge-and-trap extraction method for volatile organic compounds (VOCs).

1.1 This SOP integrates additional method guidance provided through EPA memoranda, articles by reputed USACE researchers such as Mr. Alan D. Hewitt, and draft methods from EPA regional offices. These are referenced in Section 11 of this SOP.

1.2 Parts of this SOP are based on Draft Method 5035A, which applies the conclusions made by USACE researcher Mr. Alan D. Hewitt. An excerpt from a peer-reviewed article by Mr. Hewitt is reproduced verbatim: "This study showed that the frozen storage of samples in a VOA vial or EnCore Sampler was consistent with the intent of the new guidance, and in general, 90% or greater of the concentrations were retained over a 14-day period for five different soil matrices. For several reasons, this method of sample preservation appears to be better suited for VOCs in soil matrices than acidification."

1.3 Samples are preserved either by freezing or by adding preservatives such as methanol (for high level samples) or sodium bisulfate solution (for low level samples) followed by refrigeration.

1.4 Analytical Management Laboratories (AML) personnel, AML contractors, AML clients, etc may perform these procedures in the field. When performed by non-AML personnel, this SOP shall be interpreted as a guidance document for accurate sampling and analysis for VOCs. Simpler guidance documents are also available for use in the field.

1.5 Purge and trap procedures are included in the SOPs that deal with sample analysis. Analytical procedures will include procedures that are based on SW-846 Methods 8260, 8021, 8015, etc. This SOP should be used in conjunction with other AML SOPs used for sample analysis.

1.6 This SOP revision replaces Revision A, which integrated and replaced three older 5035-based AML SOPs each dealing with one of the following: (1) sample collection, (2) sample preservation, and (3) sample analysis.

1.7 Implementation of this SOP will require good coordination between personnel collecting samples in the field, personnel preparing sampling kits, persons receiving samples at the laboratory and laboratory personnel performing VOCs analysis. It will also require coordination between client and laboratory project managers during project planning and work plan development.

1.8 This SOP provides several options or plans for sample collection and preservation that are summarized in Section 2 and described in detail in Section 8. These options are necessary due to client preferences for sampling and preservation. Plan A, which is cost effective, technically sound, and the simplest to implement both in the field and in the laboratory is the recommended option.

2.0 SUMMARY OF METHOD

2.1 The SW-846 method and related guidance documents allow several options for sample collection and preservation. Samples are collected for the following: (1) analysis by the low concentration soil method; (2) analysis by high concentration soil method; (3) analysis by high concentration oily waste method; and (4) determination of percent solids and/or acid reactivity (sampling syringe or 2-Oz jars). Options for sample preservation include the following: (1) field preservation and (2) preservation at the laboratory.

2.2 Sampling kits provided by the laboratory and procedures employed (Plans A to D and Plans E to F) depend on the chosen option. The samples remain hermetically sealed (closed system) from the time of collection for Plans A to D. However, this is not true for plans (E and F) that involve proprietary EnCore® samplers. Plans that involve EnCore® samplers are not recommended by AML due to their high cost and potential loss of VOCs during sample transfer and preservation at the laboratory. A summary of each plan is shown below:

2.2.1 Plan A - This plan consists of collecting soil samples in three (3) pre-weighed and labeled 40-ml VOA vials, each containing a 0.125" magnetic stirrer. Upon receipt by the laboratory, the vials are weighed and stored frozen at temperatures that are less than -12°C until sample analysis.

2.2.2 Plan B - This plan consists of collecting soil samples in three (3) pre-weighed and labeled 40-ml VOA vials containing 0.125" magnetic stirrers. Upon receipt by the laboratory, the vials are weighed, sodium bisulfate preservative is added through the septum to two of the vials and methanol is added to the third using a syringe. The vials are stored at 4°C until sample analysis.

2.2.3 Plan C - This plan consists of collecting soil samples in three (3) pre-weighed and labeled 40-ml VOA vials containing 0.125" magnetic stirrers. Two of the vials will also contain the sodium bisulfate solution (low soil method) preservative and the third vial will contain methanol (high soil method). Upon receipt by the laboratory, samples are weighed, and stored at 4°C until sample analysis.

2.2.4 Plan D - This plan consists of collecting soil samples in three (3) pre-weighed and labeled 40-ml VOA vials containing 0.125" magnetic stirrers. Immediately after adding soil samples, the field sampler will cap and weigh each vial and record soil weights. Immediately thereafter, the field sampler will add sodium bisulfate preservative to two of the vials and methanol to the third. The field sampler will cap and weigh each of the vials again and record them. Upon receipt by the laboratory, the vials are weighed again for quality assurance and stored at 4°C until sample analysis. This is the most complicated option, which may result in the rejection of some or all field samples by the laboratory. Samples may be rejected if leakage is suspected based on weight differences. Samples may also be rejected if the weights recorded in the field are deemed unreliable.

2.2.5 Plan E - This plan consists of collecting soil samples in three (3) 5-g EnCore® cartridges. Upon receipt by the laboratory, the cartridges are stored frozen at temperatures that are less than -12°C until sample analysis. Just prior to analysis, the cartridges are thawed, opened by analyst, contents transferred to three pre-weighed and labeled 40-ml vials containing 0.125" magnetic stirrers. The vials are capped, weighed, soil weights recorded and samples analyzed using appropriate (low or medium) methods.

2.2.6 Plan F - This plan consists of collecting soil samples in three (3) 5-g EnCore® cartridges. Upon receipt by the laboratory, the cartridges will be opened by the laboratory sample custodian, the contents transferred to three pre-weighed and labeled 40-ml vials containing 0.125" magnetic stirrers. The vials are capped, weighed and the soil weights recorded. Immediately thereafter, the sample custodian will add sodium bisulfate preservative to two of the vials and methanol to the third. The vials are stored at 4°C until sample analysis.

2.3 Additional sample aliquots are collected in the field for the determination of percent solids. These aliquots are also used for characterizing samples for acid reactivity, so that appropriate

preservatives may be added to the samples that are preserved at 4°C. Two methods are used for sample collection:

2.3.1 Syringe sampling - When procedures described in Sections 2.2.1 to 2.2.4 (Plans A to D) are used, an additional aliquot is collected by simply filling the syringe with the soil sample. Additional soil samples may also be collected in 2-Oz jars for percent solids determination.

2.3.2 EnCore® sampling - An additional soil sample is collected in 2-Oz jars when procedures described in Sections 2.2.5 to 2.2.6 (Plans E to F) are employed.

3.0 INTERFERENCES

3.1 Solvents commonly used in the field (e.g. acetone, hexane, isopropanol, etc.) for decontamination, fuel (e.g. gasoline, diesel, etc.) for operating cars, trucks, generators, etc. and impurities associated with sample preservatives (sodium bisulfate, water, methanol, etc.) are major sources of contamination. Review of method blank, trip blank, reagent blank, and field blank data should give insight into the types and sources of the contamination. If corrective actions are not successful in resolving contamination problems, an explanation of the problem should be included in reports.

3.2 Field cross-contamination may occur when samples with low levels of target analytes are collected from sampling locations that are adjacent to those containing high levels of the same target analytes.

3.3 A trip blank is analyzed to check for contamination during sample shipment and storage procedures. Trip blanks that originate from the laboratory are prepared by filling two 40-ml VOA vials containing acid preservatives with Type I water from the USFilter Purelab Plus system (See AML SOP Q-Water-RA for details). Trip blanks are usually prepared when the laboratory receives client request for sample shuttle. The laboratory should use the same water source for the analysis of method blanks.

3.4 Trip blank samples are delivered to the field samplers, who should store them with unused VOC sampling kit, and ship them in sample coolers that contain the collected field VOC samples.

3.5 Disposable cut plastic syringes, Encore ™ samplers and 40-ml VOA vials free of target VOC compounds for collecting samples in the field. Pre-cleaned vials, syringes dedicated for each sample, Encore ™ samplers, which cannot be re-used are used to minimize cross-contamination and/or carryover from one sampling location to another.

3.6 Syringe and/or cartridge holders should be decontaminated at the desirable frequency to minimize contamination and/or carryover.

3.7 Soil samples may contain lime, carbonates, bicarbonates, etc., which may react with the acid preservative (sodium bisulfate) used for preservation. The gas evolved may purge VOCs from the sample. Slow reactions may result in gas build-up within the vial, which may result in exploded sample vials. Field preservation of soil samples should be preceded by careful evaluation of soil characteristics.

4.0 APPARATUS

4.1 Sampling syringe holder from (e.g. Powerstop Handle® from US Analytical, Inc. (USAI, catalogue #AB-102)

4.2 Disposable cut-off syringes (e.g. EasyDraw Syringe® from USAI, catalogue #AB-101)

4.3 VOA vials, 40-mL, screw-cap, PTFE lined, septum-sealed sample containers that mate with purge and trap autosamplers (QEC catalogue #3112-40ml and 3122-40ml for amber vials)
4.4 PTFE coated magnetic stirring bars (0.125") to fit inside 40-mL VOA vials (QEC catalogue #5035-1008, 25 to a package)
4.5 Pre-weighed VOA vials with stir bars as described in Sections 4.3 and 4.4 (QEC catalogue # 3112-40ST and 3122-40ST for amber vials)
4.6 Pre-weighed VOA vials with stir bars and sodium bisulfate preservative (QEC catalogue # 3112-40NahST and 3122-40NahST for amber vials)
4.7 Pre-weighed VOA vials with stir bars and methanol preservative (QEC catalogue # 3112-40CH3T and 3122-40CH3T for amber vials)
4.8 2-Oz. clear wide mouth sample jars with PTFE lined screw caps
4.9 Field portable top-loading balance capable of weighing to 0.01 g
4.10 EnCore® sampling cartridges, 5-g capacity, QEC catalogue #0535-0004
4.11 EnCore® sampling cartridges, 25-g capacity, QEC catalogue #0535-0005
4.12 EnCore® T-Handle, QEC catalogue #0535-0006
4.13 EnCore® Extrusion Tool, QEC catalogue #0535-0007
4.14 Foam pouches to hold approximately three 40-ml VOA vials
4.15 Syringes, 5-mL, gas-tight with shut-off valve
4.16 Syringe needles, 22-gauge
4.17 Stainless steel spatulas
4.18 Disposable glass vials (5-ml) with screw caps
4.19 Disposable test tubes, 13x100 mm
4.20 Disposable Pasteur pipettes
4.21 Class A Volumetric flasks (500-mL and 250-mL) with ground glass stoppers
4.22 Other sampling tools as specified in the field sampling plan (FSP) or the sampling and analysis plan (SAP)

5.0 REAGENTS AND PRESERVATIVES

5.1 It is assumed that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where available. Any other reagents must demonstrate high purity without lessening the accuracy of the determination.
5.2 Organic analyte-free reagent water: Type I water from the USFilter Purelab Plus system (See AML SOP Q-Water-RA for details.
5.3 Methanol, purge-and-trap quality.
5.4 Hexadecane, ACS Reagent Grade.
5.5 Target analyte-free polyethylene glycol (PEG), ACS reagent grade.
5.6 Sodium bisulfate ($NaHSO_4$), ACS reagents grade. Bake it at 100°C overnight, allow to cool inside a desiccator and store it in an organic solvent free area prior to use.
5.7 Prepare a 20% $NaHSO_4$ solution by weighing 100 g of $NaHSO_4$ into a 500-mL volumetric flask and making up the volume to the 500-mL mark with reagent water. Prepare 250-mL or other lower volumes by appropriate scaling of the reagents. The term "acid preservation" in this document is synonymous with preservation with 20% $NaHSO_4$ solution.

6.0 HEALTH AND SAFETY

6.1 Reagents such as sodium bisulfate solutions are acidic and corrosive. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

6.2 Reagents such as methanol are flammable and highly toxic, if ingested. Gloves and safety glasses should be worn to prevent contact with skin and eyes.
6.3 Samples may contain high levels of VOCs and other organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.
6.4 Sample vials that are known to contain high concentrations of vapors in the headspace should be opened in a hood to minimize exposure to organic vapors.
6.5 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to organic vapors.

7.0 WASTE DISPOSAL

7.1 AML personnel should refer to AML SOP on waste disposal for procedures dealing with the disposal of unused preservatives, sample containers, etc. Other sampling personnel should refer to the FSP, SAP, or other applicable guidance document.

8.0 PROCEDURES

Several options exist for soil/sediment sample collection and preservation using SW-846 Method 5035. These options are labeled as plans in this document. The procedures to be followed for each option or plan are described below in Sections 8.1 to 8.6. The procedure for oily waste samples is described in Section 8.7:

8.1 **Plan A** (Section 2.2.1) - ***This is the preferred option.*** This option will be used by AML as the default option, when other options are not specified in the FSP or SAP. Balances and preservatives are not required in the field for this option. Under this option, samples are collected in the field and shipped immediately so that they are received by the laboratory within 48-hours of collection. Upon receipt, the samples are examined, weighed, logged in and stored frozen at -12°C until analysis.

8.1.1 For each soil or sediment sample to be collected, assemble a kit, which consists of the following: three pre-weighed and labeled 40-ml VOA vials, each containing a 0.125" magnetic stirrer (Section 4.5); one EasyDraw Syringe® (Section 4.2), and one foam pouch (Section 4.14). For each sampling event, assemble a kit, consisting of the following: one Powerstop Handle® syringe holder (Section 4.1); cooler(s) of appropriate size; trip blank(s) as described in Sections 3.3 and 3.4; and zip-lock bag containing chain of custody forms, sample labels, custody seal, etc.

8.1.2 The syringe holder will have settings for collecting 5-gram, 10-g or 13-gram samples for different analyses. Remove cap from the syringe. Insert the syringe into one of the three (heavy, medium, and light) 5-g positions. See Appendix 1 for guidance from the manufacturer. Push the syringe into the representative soil being sampled until the soil column inside the syringe has forced the plunger to the stop point (top of the syringe holder). Remove the syringe from the holder, wipe the exterior of the syringe with a paper towel, insert syringe into one of the open pre-weighed 40-ml vials and eject sample into it. Avoid getting dirt on the threads of the 40-ml vial. Do not remove the stirring bar from the vial. Wipe the vial threads if necessary, cap vial immediately, and store at 4°C.

8.1.3 Collect two additional 40-mL vials for each sample using procedures described in Section 8.1.2. Use the same syringe for collecting additional vials.

8.1.4 Insert the syringe into the lowest (13-g) position. Collect an additional aliquot for the determination of percent solids (see Section 2.3.1) and cap syringe.

8.1.5 Upon receipt at the laboratory, the sample custodian will assign a laboratory sample number for each sample and labels such as A, B, and C to each of the three 40-ml vials and document them in Form-A (Appendix 2). The sample custodian will copy into Form-A the tare weight of each vial that has been pre-determined and recorded on the vials. The sample custodian will determine the weight of the vial plus sample to two decimal places and record it on Form-A. The sample weight is the difference between the two weights.

8.1.6 Tighten the cap on the vials, if necessary and store them at -12°C in a freezer. Enter "F" for the type of preservation for all the vials in Form A. ***NOTE: The tightening step is necessary, since the rubber seal may shrink when the vials are frozen. Vapor-tight seal must be maintained during storage.***

8.1.7 Label the syringe appropriately and store them at 4°C in the walk-in cooler along with other project samples.

8.1.8 Immediately before analysis, the analyst will allow one (or more vials at analyst's discretion) of the vials to thaw and warm up to room temperature. The analyst will perform analysis as described in the respective analytical method without the addition of preservatives for low level method. The vials will never be opened in the laboratory.

8.1.9 If the results of the initial analysis indicate the need for medium level analysis, sample contained in one of the three vials will be preserved with methanol. See Section 8.2.5 for methanol preservation. Enter date of preservation and type ("M" in addition to "F") for methanol extraction/preservation of the affected sample vials in Form A. Include volume of methanol in Form A. The default volume is 5.0-ml. Volumes (ml) that are equal to the wet sample weight (g) may be used at analyst's discretion. See SOP for the respective analytical method for medium level analysis.

8.2 **Plan B** (Section 2.2.2) - This option (Plan B) is identical in sample collection to Plan A. Under this option, samples are collected in the field and shipped immediately so that they are received by the laboratory within 48-hours of collection. Upon receipt, the samples are examined, weighed, logged in, preserved and stored at 4°C until analysis.

8.2.1 See Sections 8.1.1 to 8.1.4.

8.2.2 See Section 8.1.5. Record weights in Form-B (Appendix 3).

8.2.3 Using a clean spatula, transfer approximately 1 g of the sample from the syringe (or the 2-OZ jar) to a labeled, clean 5-mL screw cap vial. Add 1-mL of the $NaHSO_4$ reagent. Seal the vial tight with a screw cap, shake and set aside. Observe for effervescence and record it on the "Comments-lab use only" column in Form-B. After approximately 1 hour, open the vial looking for pressure build-up. Record date and any observations such as "pressure", "effervescence", etc. in Form-B. If neither effervescence nor pressure build-up is noticed, two of the vials for each sample can be preserved with acid for low concentration analysis. **NOTE:** If the sample reacts with $NaHSO_4$, preservation of the affected samples with this reagent should be abandoned. Use Plan A (Sections 8.1.6 and 8.1.7) for sample preservation and analysis. Enter "F" for the type of preservation for the affected sample vials in Form B.

8.2.4 $NaHSO_4$ preservation of samples for low level analysis should be performed by injecting 5-mL of the $NaHSO_4$ solution (Section 5.7) into two 40-mL VOA vials for each sample using a 5-mL syringe equipped with a sharp 22-gauge needle. Do ***NOT*** use heavier gauge needles or needles that are damaged or defective for preserving the integrity of the septum seal. Record the preservative (A) and its volume in Form-B. Store vials at 4°C until the analyses are complete.

8.2.5 Methanol preservation of samples for medium or high level analysis should be performed by injecting 5-mL of the purge and trap grade methanol (Section 5.3) into the third 40-mL VOA vial using a 5-mL syringe equipped with a sharp 22-gauge needle. Do ***NOT*** use heavier gauge needles or needles that are damaged or defective. Record the preservative (M) and its volume in Form-B. The default volume for methanol is 5.0-ml. The sample custodian may use volumes (ml) that are equal to the wet sample weight (g) if directed by the analyst. The analyst may open the 40-ml vial and transfer the methanol extract to an appropriately labeled 5-ml vial (Section 4.18) for convenience. Store vials at 4°C until the analyses are complete.

8.2.6 Immediately before analysis, the analyst will allow the vials to warm up to room temperature and perform analysis as described in the respective analytical method. The $NaHSO_4$ preserved vials will never be opened in the laboratory.

8.3 **Plan C** (Section 2.2.3) - This option (Plan C) is identical in sample collection to Plan A. Under this option, samples are collected into vials containing preservatives. Upon receipt, the samples are examined, weighed, logged in, preserved and stored at 4°C until analysis.

8.3.1 For each soil or sediment sample to be collected, assemble a kit consisting of the following: two pre-weighed and labeled 40-ml VOA vials, each containing a 0.125" magnetic stirrer and $NaHSO_4$ preservative (Section 4.6); one pre-weighed and labeled 40-ml VOA vial containing a 0.125" magnetic stirrer and the methanol preservative (Section 4.7); one EasyDraw Syringe® (Section 4.2); and one foam pouch (Section 4.14). For each sampling event, assemble a kit, consisting of the following: one Powerstop Handle® syringe holder (see Section 4.1); cooler(s) of appropriate size; trip blank(s) as described in Sections 3.3 and 3.4; and zip-lock bag containing chain of custody forms, Form-C (Appendix 4), sample labels, custody seals, etc. The kit should also contain a portable balance (Section 4.9) and additional glassware to perform field reactivity tests (Sections 4.17 to 4.20). The laboratory is not responsible for providing these items (Sections 4.9 and 4.17 to 4.20) to non-AML personnel.

8.3.2 Perform acid reactivity tests for each sample in the field as described in Section 8.2.3. (**NOTE:** Have additional vials without preservatives (Section 4.5) ready for sample preservation, if acid preservation needs to be abandoned. The laboratory does not provide additional vials (Section 4.5) to non-AML personnel unless specifically requested by the client. Additional charges will apply. Plan D (Section 8.4), which is even more complex to perform in the field should be considered for sample collection as an alternative.) If acid reactivity is not a problem, see Section 8.1.2 for sample collection. Make sure that the preservative does not splash out from the vial during the addition of soil. Copy the tare weight (VP, in grams to two decimal places) of the vial (includes the stir bar, label, cap, and the preservative) into Form-C (Appendix 4). Weigh the vial (with cap) plus sample (FW) to two decimal places in the field and record the weights in Form-C. Store sample vials at

4°C until samples are ready for shipment. Operations described in this section are performed in the field.

8.3.3 See Section 8.1.3. See additional guidance as described in Section 8.3.2.

8.3.4 See Section 8.1.4.

8.3.5 Upon receipt of samples at the laboratory, the sample custodian will assign a laboratory sample number for each sample and labels such as A, B, and C to each of the three 40-ml vials. The sample custodian will wipe the vials dry and determine the weight of each vial plus sample (LW) to two decimal places for quality assurance purposes and record them on Form-C submitted by the field samplers. If the difference (FW-LW) between the weights recorded in the field and the laboratory is greater than 0.2-g, the integrity of the sample (due to potential leaks during shipment) or field gravimetric measurements may be suspect. Corrective actions, which normally involve re-sampling may be necessary for the affected samples. Contact the client Project Manager and/or the sampler immediately for guidance. The laboratory will adhere to decisions made by the Project Manager. Sample weights (FW-VP) used in analyses are based on field gravimetric measurements. Store vials at 4°C until the analyses are complete.

8.3.6 See Section 8.2.9. The $NaHSO_4$ preserved vials will never be opened in the laboratory. The analyst may open the 40-ml vial intended for medium level method and transfer the methanol extract to an appropriately labeled 5-ml vial (Section 4.18) for convenience. Store vials at 4°C until the analyses are complete.

8.4 **Plan D** (Section 2.2.4) - This option is similar to Plan C. Balances and preservatives are required in the field for this option. Under this option, samples are collected into pre-tared vials, weighed, and preservatives are added into the vials in the field. The vials are re-weighed in the field and samples are shipped to the laboratory. Upon receipt, the samples are examined, re-weighed for quality assurance, logged in, preserved and stored at 4°C until analysis.

8.4.1 See Sections 8.1.1. The kit should also contain copies of Form-D (See Appendix 5) and preservatives (Sections 5.3 and 5.7) that will be used in the field. The kit should also contain a portable balance (Section 4.9) and additional glassware to perform field reactivity tests (Sections 4.17 to 4.20). The laboratory is not responsible for providing these items (Sections 4.9 and 4.17 to 4.20) to non-AML personnel. Perform acid reactivity test in the field as described in Section 8.2.3.

8.4.2 Perform sample collection as described in Sections 8.1.2 and 8.1.3. Keep the samples dry and at room temperature during gravimetric measurements, which are described in the following sections.

8.4.3 Copy the tare weights (two decimal places) of the vials (V) into Form-D (Appendix 5). Tare weights of the vials may need to be determined before addition of samples, if they have not been pre-determined. Determine the weights of the vial and the sample (VS) to two decimals and record them in Form-D. Calculate sample weights (S) using the equation, S = VS-V and record it in Form D.

8.4.4 Remove the vial cap and dispense 5-ml of the 20% $NaHSO_4$ solution preservative (Section 5.7) into two of the vials. Replace the caps immediately and re-weigh the vials (FW) containing sample and preservatives. The exterior of the vials should be wiped dry before gravimetric measurements are performed. Record weights to two decimal places in Form D. Repeat this procedure for the third vial using methanol (Section 5.3) as the preservative. The sampler instead of using the default volume

(5-ml) may dispense volumes (ml) of methanol that are equal to the wet sample weight (S in grams). Store sample vials at 4°C until samples are ready for shipment. These operations are performed in the field.

8.4.5 When samples are received in the laboratory, they are handled as described in Section 8.3.5 with the exception that Form-D from the field is used to document laboratory findings.

8.4.6 See Section 8.3.6

8.5 **Plan E** (Section 2.2.5) - This option (Plan E) is similar in sample collection to Plan A. Under this option, samples are collected in specially designed zero head space cartridges in the field and shipped immediately so that they are received by the laboratory within 48-hours of collection. Upon receipt, the samples are examined, weighed, logged in, and stored at -12°C until analysis.

8.5.1 The EnCore® samplers (Sections 4.10 and 4.11) are specially designed 2-piece (coring body and cap) cartridges that are provided in sealed pouches. The body of the samplers consists of a coring body, a plunger bottom with a rubber O-ring for seal, a plunger rod, slots for mating with the T-Handle/Extruder (Sections 4.12 and 4.13) and a ridge for securing the cap. The cap has an O-ring for seal and two locking arms with grooves that mate with the ridge on the coring body. See Appendix 6 (2-pages) for a description of the sampler and sampling procedures.

8.5.2 This option involves opening the cartridges in the laboratory (potential loss of VOCs) for the transfer of samples into 40-ml VOA vials that are described for Plans A, B, and D. The vials will be capped for weighing and opened again for the addition of preservatives. Although this option is available under method 5035, it does not meet the definition for a "closed system from the time of sample collection", which is one of the technical requirements to minimize subsequent loss of VOCs from the sample. This option will also add a minimum of $25 per sample in sample collection supply costs, which may be a big burden for some laboratory clients.

8.5.3 For each soil or sediment sample to be collected, assemble a kit, which consists of the following: three 5-g EnCore® samplers or cartridges (See Section 4.10) and one-quart zipper bag (Section 4.22). For each sampling event, assemble a kit, consisting of the following: one EnCore® T-Handle (Section 4.12)or a T-Handle/Extruder combo (Section 4.13); cooler(s) of appropriate size; trip blank(s) as described in Section 3.3; and a zip-lock bag containing chain of custody forms, sample labels, custody seal, etc. The 25-g EnCore® samplers or cartridges (See Section 4.11) may be used for special tests other than VOCs.

8.5.4 See Appendix 6 for guidance from the manufacturer. Hold coring body (See figure in Appendix 6) and push plunger rod until small O-ring rests against tabs. This will assure that plunger moves freely. Depress locking lever on the EnCore® T-Handle. Place coring body, plunger end first, into open end of T-Handle, aligning the two slots on the coring body with the two locking pins in the T-Handle. Twist coring body clockwise to lock pins in slots. Check to ensure Sampler is locked in place. Turn T-Handle with T-up and coring body down. This positions plunger bottom flush with bottom of coring body. Sampler is now ready for use.

8.5.5 Using the T-Handle, push Sampler into the representative soil being sampled until the soil column inside the Sampler has forced the plunger to the stop point. When the coring body is full, small O-ring will be centered in T-Handle viewing hole.

Remove the Sampler from the soil, wipe the excess soil from coring body exterior with a paper towel, and cap coring body while it is still on T-Handle. Push cap over flat area of ridge and twist to lock cap in place. Cap must be seated to seal sampler. See diagrams in Appendix 6 for guidance. Remove the capped Sampler by depressing locking lever on T-Handle while twisting and pulling Sampler from T-Handle. Lock plunger by rotating extended plunger rod fully counterclockwise until wings rest firmly against tabs. Attach completed circular label (from EnCore® Sampler bag) to cap on coring body. Return full EnCore® Sampler to zipper bag. Seal bag and store them in the field at 4°C.

8.5.6 Collect sample into two additional Samplers for each soil sample using procedures described in Sections 8.5.4 and 8.5.5. Store the three smaller bags containing one Sampler each into a larger zipper bag.

8.5.7 Collect an additional soil sample into a 2-Oz jar for the determination of percent solids, acid reactivity and other sample characteristics in the laboratory. Store samples in the field at 4°C.

8.5.8 Upon receipt at the laboratory, the sample custodian will assign a laboratory sample number for each sample and labels such as A, B, and C to each of the three Samplers. The sample custodian will prepare Form-E as part of the log-in procedure.

8.5.9 The sample custodian will store Samplers at -12°C in a freezer.

8.5.10 The laboratory sample custodian will prepare a kit consisting of three pre-weighed and labeled 40-ml VOA vials, each containing a 0.125" magnetic stirrer (See Section 4.5) for each sample that requires analysis. The VOA vials should be stored at room temperature.

8.5.11 The samples contained in the 2-Oz jars will be labeled appropriately and stored at 4°C.

8.5.12 Immediately before analysis, the analyst will allow the one or more Samplers to thaw and warm up to room temperature. The soil samples are transferred to 40-ml VOA vials (Section 4.5) using procedures described below.

8.5.13 Copy the tare weights (two decimal places) of the vials into Form-E (Appendix 7). Tare weights of the vials may need to be determined before addition of samples, if they have not been pre-determined. Use a pair of pliers to break locking arms on cap of the EnCore® Samplers. Do not remove cap at this time. Use the T-Handle/Extrusion tool (Sections 4.12 and 4.13) to remove samples from the Sampler. Depress locking lever on Extrusion Tool and place Sampler, plunger end first, into open end of Extrusion Tool, aligning slots on the coring body with pins in Extrusion Tool. Turn coring body clockwise until it locks into place. Release locking lever. Rotate and gently push Extrusion Tool plunger knob clockwise until plunger slides over wings of coring body. Note that the plunger will not rotate further, when it is properly positioned. Hold extrusion tool with capped Sampler pointed upward so soil does not fall out when cap is removed. To release soil core, remove cap from Sampler, insert Sampler into open end of pre-tared 40-ml vial (Section 4.5) and push down on plunger knob of Extrusion Tool. Avoid getting dirt on the threads of the 40-ml vial. Do not remove the stirring bar from the vial. Wipe the vial threads if necessary and cap vial immediately. Determine the weight of the soil and the vial and record it in Form-E.

8.5.14 Perform analysis as described in the respective analytical method. Use additional Samplers and prepare them as described in Sections 8.5.12 and 8.5.13 for low level re-analysis.

8.5.15 Use the following procedure to perform methanol preservation of samples for medium level analysis. After determining the soil weight (Section 8.5.12 and 8.5.13) remove the cap and add 5-mL of purge and trap grade methanol (Section 5.3) into the third 40-mL VOA vial using a 5-mL syringe. Record the preservative (M) and its volume in Form-E. The methanol extracts may be transferred to appropriately labeled 5-ml vials (Section 4.18) by the analyst for convenience. Store vials at 4°C until the analyses are complete.

8.6 **Plan F** (Section 2.2.6) - This option (Plan F) is similar in sample collection to Plan E. Under this option, samples are collected in specially designed zero head space cartridges in the field and shipped immediately so that they are received by the laboratory within 48-hours of collection. Upon receipt, the samples are examined, logged-in, transferred into 40-ml VOA vials, preserved, and stored at 4°C until analysis.

8.6.1 See Sections 8.5.1 to 8.5.7 for field activities.

8.6.2 See Sections 8.5.8 to 8.5.13 and 8.5.15 for log-in activities with the exception that the sample custodian will perform sample preservation functions that are described for the sample analyst. The laboratory sample custodian will log-in, perform acid reactivity and solubility tests, and preserve samples within 48-hours of sample collection as described below in detail. Use Form-F (Appendix 8) to document sample weights, observations, etc.

8.6.3 See Section 8.2.3 and perform reactivity/solubility tests using an aliquot of the sample from the 2-Oz jar.

8.6.4 The sample custodian will perform $NAHSO_4$ acid preservation of samples contained in two 40-ml vials for low level analysis. Copy the tare weights (two decimal places) of the vials into Form-F. Tare weights of the vials may need to be determined before addition of samples, if they have not been pre-determined. After transferring the soil from the EnCore® Samplers, determine and record the soil weight (to two decimal places) in Form F. Remove the cap and add 5-mL of the 20% $NAHSO_4$ solution (Section 5.7). Record the preservative (A) and its volume in Form-F. Store vials at 4°C until the analyses are complete.

8.6.5 The sample custodian will perform methanol preservation of the sample contained in the third 40-ml vial for medium level analysis. Copy the tare weights (two decimal places) of the vials into Form-F. Tare weights of the vials may need to be determined before addition of samples, if they have not been pre-determined. After transferring the soil from the EnCore® Samplers, determine and record the soil weight (to two decimal places) in Form F. Remove the cap and add 5-mL of the purge and trap grade methanol (Section 5.3) into the third 40-mL VOA vial using a 5-mL syringe. Record the preservative (M) and its volume in Form-F. Store vials at 4°C until the analyses are complete.

8.6.6 See Section 8.2.9.

8.7 **Oily Waste Samples** - For wastes that appear to be oily and water immiscible, collect a 1-2 gram sample into one of the pre-weighed 40-mL VOA vials (Section 4.5) using either a spatula or a Pasteur pipette in addition to collecting two 5-g samples in 40-mL VOA vials. Collect an additional jar for percent solids determination and sample characterization. Check

solubility of the waste in hexadecane (Section 5.4) and polyethylene glycol (Section 5.5) in addition to methanol using procedures similar to those described in Section 8.2.6. for determining acid reactivity. Do not acid-preserve samples that are soluble in hexadecane or methanol. Refer to SW-846 Method 3585 for waste dilution procedures for volatile organics analysis.

9.0 CALCULATIONS

9.1 Sample weights for this method are recorded in special forms (Forms A to F). Detailed calculations other than additions and subtractions are not required for this method.

10.0QUALITY CONTROL

10.1Sampling equipment (syringe holder, spoons, etc) must be kept clean, making sure that they are not contaminated with organic solvents. If solvents (e.g. methanol) are used to clean the syringe holder, it must be dried thoroughly prior to use.

10.2 Indelible ink markers shall be used to record field assigned sample numbers on each vial for each sample collected. Record the vial number and the sample number on the Form provided and/or in the field logbook. Record sample numbers on sample pouches that contain EnCore® cartridges and seal them.

10.3 Collect one extra set of containers for each 20 samples or less for field duplicate analysis. An additional replicate may be necessary if samples need to be sent to an external laboratory for QA split sample analysis.

10.4 Collect two extra sets of samples for matrix spike (MS) and matrix spike duplicate (MSD) analysis for each 20 project samples or less. The sample designated for replicate analyses may be chosen for MS and MSD analyses.

10.5 Refer to an approved sampling and analysis plan (when available) to determine the appropriate number of quality control samples that needs to be collected.

11.0 REFERENCES

11.1Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5035, Closed-System purge-and-trap and Extraction for Volatile Organics in Soil and Waste Samples, Revision 0, December 1996.

11.2Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5035A, Closed-System purge-and-trap and Extraction for Volatile Organics in Soil and Waste Samples, Draft Revision 1, July 2002.

11.3 Clarification Regarding Use of SW-846 Methods, EPA Memorandum, Office of Solid Waste, Washington, D.C., August 7, 1998, page 1 to 3, Attachment 1, page 1 to 3, and Attachment 2, page 3 to 6.

11.4 Hewitt, Alan D. (1999) Frozen Storage of Soil Samples for VOC Analysis, Environmental Testing and Analysis, Volume 8, Number 5, p 18 to 25 and p 46.

11.5Draft Guidance for SW-5035 implementation, EPA, Region V, September 2000.

11.6Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8260B, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), December 1996.

11.7 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

11.8 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.

11.9 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5000, Sample Preparation for Volatile Organic Compounds, December 1996.

11.10 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5021, Volatile Organic Compounds in soils and other solid matrices using equilibrium headspace analysis, December 1996.

11.11 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5030A, Purge and Trap, July 1992.

11.12 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5030B, Purge and Trap for Aqueous Samples, December 1996.

11.13 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3585, Waste Dilution for Volatile Organic Compounds, December 1996.

11.14 Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, EPA, 40 CFR Part 136, Method 624: Purgeables, October 1984.

11.15 Methods for the Determination of Organic Compounds in Drinking Water, Supplement II, EPA/600/R-92/129, Method 524.2, Measurement of Purgeable Organic Compounds in Water by Capillary column Gas Chromatography / Mass Spectrometry, August 1992.

11.16 AML Standard Operating Procedures, AML-5035-SA1.

11.17 AML Standard Operating Procedures, AML-5035-SC1.

11.18 AML Standard Operating Procedures, AML-5035-SP1.

11.19 AML Standard Operating Procedures, Q-WMADISD-RA.

Table 1
List of Available Appendices
(Not included in this document)

Appendix Number	Appendix Topic
1	Sample collection with EasyDraw Syringe® and PowerStop Handle® Instructions from the Manufacturer for Sample Collection
2	Form A – Soil sample collection and preservation Record for AML Plan A
3	Form B – Soil sample collection and preservation Record for AML Plan B
4	Form C – Soil sample collection and preservation Record for AML Plan C
5	Form D – Soil sample collection and preservation Record for AML Plan D
6	Sample Collection with EnCore® Sampler and T-Handle/Extrusion Tool Instructions from the Manufacturer for Sample Collection
7	Form E – Soil sample collection and preservation Record for AML Plan E
8	Form F – Soil sample collection and preservation Record for AML Plan F

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices. The appendices may be available in hardcopy form or as portable document electronic files (pdf).

STANDARD OPERATING PROCEDURES

No. O-VOCs-GCMS-RC

VOLATILE ORGANIC COMPOUNDS IN WATER AND SOIL BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) (EPA Methods)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY / MASS SPECTROMETRY (GC/MS)

1.0 SCOPE AND APPLICATION

1.1 This Analytical Management Laboratories (AML) standard operating procedure (SOP) is based on EPA SW-846 Method 8260B, Method 5030C, Draft Method 5035A, and other EPA methods such as 624, 524.2, OLM03.0, OLM04.0, etc. This SOP also incorporates guidance from USACE documents such as Appendix I, of EM200-1-3. Words and acronyms such as method and SOP are used interchangeably in this document.

1.2 This SOP is used to determine volatile organic compounds in a variety of matrices including water, air, soil and waste. Water samples will include groundwater, surface water, drinking water, and wastewater. This method is applicable to nearly all types of samples. The compounds that can be determined by this method are in Table 1.

1.3 This method can be used to quantitate most volatile organic compounds that have a boiling point below 200°C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included; however, they will have a higher quantitation limit due to poor purging efficiency. Table 1 lists the analytes and their retention times, which are subject to change depending on chromatographic conditions. Drinking water analytes shown in Table 1 may belong to one more groups: regulated compounds, trihalomethanes, and unregulated compounds.

1.4 The method quantitation limits (MQL) for each individual compound is instrument dependent. The method detection limits (MDLs) vary for each compound and it may differ for each instrument. The instruments employed at AM Laboratories are standard quadrupole 5970 and 5973 mass specific detectors (MSDs). The MQLs, lowest limits for reporting (LLRs), and MDLs will be proportionately higher for sample extracts and samples that require dilution. See Table 4 for MQLs that are normally attainable for most samples. MQLs, MDLs, and LLRs are dynamic parameters, which are subject to change.

1.5 This method is based upon the theories of purge-and-trap, gas chromatography/mass spectrometric procedures. This method is restricted to experienced analysts in the use of these theories and skilled in the interpretation of mass spectra and their use as a quantitative tool.

1.6 The procedure can employ 5-mL or 25-mL sample volumes for purge and trap extraction. The 25-mL purge is employed to analyze drinking water and most groundwater samples to meet regulatory requirements.

2.0 SUMMARY OF METHOD

2.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method. Purged sample components are trapped in a tube containing suitable sorbent materials. When purging is complete, the sorbent tube is heated and backflushed with helium to desorb trapped sample components. The analytes are desorbed directly to a capillary column before being flash evaporated to a narrow bore capillary for analysis. The column is temperature programmed to separate the analytes, which are then detected with a mass spectrometer interfaced to the gas chromatograph.

2.2 Sometimes, due to high levels of target and non-target analytes, it is necessary to extract the volatile organic compounds from the sample(s) with methanol. A portion of the methanol solution is combined with organic-free reagent water in the purge chamber. It is then analyzed by purge-and-trap GC/MS following the normal water method. Methanol extraction is not necessary when samples are preserved with methanol. See AML SOPs based on SW-846 Method 5035 and 5035A for details on sample collection and methanol preservation.

2.3 Identification of the target analytes is accomplished by comparing their mass spectra with spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitative) ion relative to an internal standard with a five-point calibration curve constructed using a similar procedure.

3.0 INTERFERENCES

3.1 Solvents commonly used in the laboratory and impurities from volatile materials are major sources of contamination. Review of blank and calibration data should give insight in to the types and sources of the contamination. Corrective action procedures should be followed when dealing with contamination. Subtracting blank values from samples is not permitted. If corrective actions are not successful in resolving blank contamination problems, an explanation of the problem should be included in the case narrative.

3.2 Interfering contamination may occur when low levels of target analytes are found immediately following the analysis of a sample containing high levels of the same target analytes. All purging apparatuses and syringes are rinsed after each sample analysis regardless of carry-over. Additional blanks may be analyzed to check for sample contamination. Screening by dilution is also employed in many instances. Generally, as a preventive maintenance measure, the purge-and-trap system is baked out at 250° C.

3.3 Methylene chloride and acetone are the most commonly found contaminants in this method. Several measures have been employed to assist in minimizing this contamination. The GC/MS volatile system is located farthest from operations that employ these solvents. The reagent water used for analysis is Type I water, prepurged if necessary to eliminate VOC contamination. All gas lines and purge plumbing are constructed of copper tubing or stainless steel.

3.4 A trip blank is analyzed to check for contamination during the shipment and storage procedures. Trip blanks that originate from the laboratory are prepared by filling two 40-ml VOA vials containing acid preservatives with Type I water from the USFilter Purelab Plus system (See AML SOP Q-Water-RA for details). Trip blanks are usually prepared when the laboratory receives client request for sample shuttle. Trip blank samples are delivered to the field samplers, who then keep the trip blank with the actual field VOC samples during sample shipment. The laboratory does not store trip blanks that have not been shipped out for sampling activities. The laboratory uses the same water source for method blanks.

3.5 The oven temperature program is designed to bake out semi-volatile hydrocarbons that may have volatilized during the purge-and-trap analysis.

4.0 APPARATUS AND MATERIALS

4.1 GC/MS and Purge/Trap Systems - Three GC/MS systems are used. Most of the analyses (water and soil) are performed using the more sensitive (Agilent 5973 Network MSD) instruments. The older instrument (HP 5970) is used for performing analyses for UST projects. The instruments and their components are described below:

4.1.1 Gas Chromatographs – Two Agilent 6890 Series II GCs (primary) and HP 5890 (backup) are used. The systems are complete with a temperature-programmable GC for splitless injection. The systems include all accessories, including syringes, analytical columns and gasses. The column flow rate remains constant throughout temperature program operation. The capillary column is directly coupled to the source.

4.1.2 Gas chromatographic columns - A 60 meter x 0.25-mm ID capillary column coated with DB-624 (J&W Scientific) is employed in both instruments. The film thickness is 1.8 um. An equivalent column may be used.

4.1.3 Purge-and-trap devices – All the instruments use Varian Archon and/or OI Model 4552 purge and trap 51-position autosamplers interfaced to Tekmar Dohrmann 3100 or 3000 sample concentrators. New models of sample concentrators (e.g. OI Eclipse Model 4660) are in evaluation. The new model concentrator may be used for analyzing aqueous samples for improving sensitivity.

4.1.4 Mass Spectrometer – Two Agilent 5973 Network MSDs (primary) and one HP 5970 (backup) mass selective detectors are used. They are capable of scanning from 35 to 400 AMU every 1-second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC. The mass spectrometers are capable of producing mass spectra for 4-Bromofluorobenzene that meets criteria listed in Table 2 when 50-ng of the BFB is injected through the GC.

4.1.5 GC/MS interface is directly coupled by inserting the column into the mass spectrometer.

4.1.6 Compressed helium gas cylinders

4.1.7 Data system and Software - It consists of several personal computers, monitors, and a HP Laserjet 4000 printer. Hewlett Packard's Enviroquant is used to allow continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the GC program. The software allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). The software also allows integrating the abundances in any EICP between specified times or scan-number limits. The most recent version of the National Bureau of Standards (NBS) mass spectral library containing 96000 compounds is used.

4.2 Microsyringes - 10, 25, 100, 250, 500 and 1000-uL.

4.3 Volumetric Flasks, class A - 10-mL, 50-mL and 100-mL with ground glass stoppers.

4.4 Balance - Analytical (Sartorius, 0.0001-g accuracy)

4.5 Revco sample storage refrigerator

4.6 Sample storage freezer

4.7 Refrigerator/Freezer to store standards

4.8 Disposable pipettes - Pasteur

4.9 Disposable 40-mL VOA vials for sample collection and for purging

4.10 Stainless Steel spatula and spoons

4.11 Magnetic stir bars
4.12 Spatula for adding ascorbic acid preservative

5.0 **REAGENTS AND STANDARDS**

5.1 All reagents used in performing analysis using this SOP shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where available. Any other reagents must demonstrate high purity without lessening the accuracy of the determination.
5.2 Organic free reagent water - All references to water in this method will be assumed to be organic free reagent water (Type I as described in Section 3.4), except when referring to field samples.
5.3 Methanol – Purge and trap quality demonstrated to be free of target analytes.
5.4 Hydrochloric acid (HCl, 1:1 v/v)
5.5 Stock Standard Solutions - Stock standard solutions are ordered through Ultra Scientific and are listed as certified solutions. The following table represents the solutions:

CATALOG NAME	CATALOG NUMBER	CONCENTRATION
VOC Mixture	DWM-588	2000-ug/mL
Ketones plus Mixture	NVM-826	2000-ug/mL
Internal/Surrogate Standard Mix	STM-541	5000-ug/ml

5.6 Working standards - Working standards are prepared to a final concentration of 100-ug/mL for water and 25-ug/ml for soil. To prepare the 100-ug/ml standard, 100-ul of the VOC mixture and 100-uls of the Ketones plus mixture are added to 1800-ul of methanol. To prepare the 25-ug/ml standard, 25-ul of the VOC mixture and 25-ul of the Ketones plus mixture are added to 1950-ul of methanol. Once the stock vials have been opened, they are to be stored in 2-mL GC vials sealed with a Teflon-lined screw cap and kept at -10° C away from any form of light. Fresh working standards are prepared on a weekly basis or when the % difference of the RRF from the check calibration standard is lowered by 15% for the gas analytes.
5.7 Internal/Surrogate working standards (ISS) for LDL volatiles (25-mls purge) - The ISS concentration employed is 10-ug/L. The Archon injects 1-ul of ISS to 25-ml of water for a final concentration of 10-ug/L. Therefore, the ISS must be at a concentration of 250-ug/ml and placed in the standards vessel on the Archon. To prepare this standard, 2.5-mL of the 5000-ug/ml solution (See Section 5.5) is added to 50-ml of methanol for a final concentration of 250-ug/ml. See Table-1 for a more details on the internal standards (Fluorobenzene, Chlorobenzene-d_5, and 1, 4-Dichlorobenzene-d_4). See Table-1 and Table 5 for details on the surrogates (Dibromofluorobenzene, 1, 2-Dichloroethane-d_4, 4-Bromofluorobenzene, and Toluene-d_8).
5.8 Internal/Surrogate working standards (ISS) for normal (5-ml purge) analyses – For the 5-ml purge and soil samples the ISS must have a concentration of 50-ug/L or 50-ug/kg. The Archon injects 1-ul of the ISS to 5-ml of water or 5-g of soil for a final concentration of 50-ug/L or 50-ug/kg.

5.9 Bromofluorobenzene (BFB) standard - A standard solution containing 50-ng/uL of BFB in methanol.

5.10 Daily Calibration Standards - Calibration standards are prepared using the working standards prepared as described in Section 5.6. Five to six daily calibration standards are prepared to generate an initial calibration curve (ICAL) as described in Table 6. These standards are made daily.

5.10.1 Water – Once the standard has been made, it is carefully poured into 40-ml VOA vials for analysis either by 25-ml purge or 5-ml purge, whichever is applicable.

5.10.2 Soil - The soil standards are made-up directly in the VOA vial itself. For example, the 10-ug/kg standard is prepared by adding 5-ml of reagent water to a 40-ml VOA vial and 2-ul of the 25- ug/ml working standard to the water in the vial. The vial is immediately sealed and ready for analysis.

5.11 Stock Initial Calibration Verification (ICV) Standards - These should be from a second source (Supelco). The stock second source used at AML is Supelco. Once the stock vials have been opened, they are to be stored in 2 ml GC vials sealed with a Teflon-lined screw cap and kept at -10° C away from any form of light. The following table represents the solutions:

CATALOG NAME	CATALOG NUMBER	CONCENTRATION
VOC Mixture	47442-U	2000-ug/mL
Ketones plus Mixture	47364	2000-ug/mL

5.12 Working ICV standards - Working standards are prepared to a final concentration of 100-ug/mL for water and 25-ug/ml for soil. To prepare the 100-ug/ml standard, 100-ul of the VOC mixture and 100-ul of the Ketones plus mixture are added to 1800-ul of methanol. To prepare the 25-ug/ml standard, 25-ul of the VOC mixture and 25-ul of the Ketones plus mixture are added to 1950-ul of methanol.

5.13 Daily ICV Standard - A fresh ICV is prepared on days in which a new initial calibration (ICAL) is performed. The concentration employed for ICV corresponds to the low-mid point (15-ug/L for 25-ml purge, 50-ug/L for 5-ml purge, and 50-ug/kg for soil). These are prepared as described in Section 5.10 and Table 6 using working ICV standards described in Section 5.12.

5.14 Daily continuing calibration verification (CCV) standard - Fresh CCVs are prepared on days in which samples are analyzed. The concentration employed for ICV corresponds to the low-mid point (15-ug/L for 25-ml purge, 50-ug/L for 5-ml purge, and 50-ug/kg for soil). These are prepared as described in Section 5.10 and Table 6 using the same source (Section 5.6) as that used for the ICAL. Prepare adequate quantities of the CCV standard to perform one or more CCV every 12 hours.

5.15 Laboratory Control Samples (LCS) and LCS Duplicate (LCSD) Samples - The preparation of LCS/LCSD samples is identical to the procedure described for the preparation of CCV standards.

5.16 Matrix Spike (MS) and MS Duplicate Samples - These spiked samples are prepared using the same standards that are used for ICAL, CCV and LCS/LCSD.

5.17 Ascorbic acid, reagent grade for preserving drinking water samples

6.0 SAMPLE PRESERVATION HANDLING & STORAGE

6.1 Groundwater, Surface water, and Wastewater samples - Standard 40-ml glass screw-cap VOA vials with Teflon lined silicone septa may be used for collecting these aqueous samples in accordance with EPA SW-846 method 5030B. Order pre-cleaned VOA vials containing the hydrochloric acid preservative directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 3 vials (4 vials may be needed for some projects) for each water sample. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in separate coolers designated for storing VOC aqueous samples.

6.2 Special 40-ml VOA vials that are used for purge-and-trap of solid samples are described in the AML SOPs based on Method 5035. The EPA SW-846 Method 5035 has several options for soil or waste sample collection. Upon receipt and log-in, samples collected on Encore cartridges, unpreserved 40-mL VOA vials, or similar devices require chemical preservation with sodium bisulfate or methanol or freezing as described in detail under other AML SOPs specific to these procedures. Samples may also be collected in 40-mL VOA vials containing preservatives. Samples should be weighed, preserved, and stored in separate coolers and/or freezers designated for storing VOC samples.

6.3 Soil samples collected with little headspace in 2-Oz or 4-Oz jars in accordance with the old 5030A (July 1992) procedure do not require chemical preservation. They should be stored in separate coolers designated for VOC samples.

6.4 Drinking water samples - The procedure described in Section 6.1 is used. Adding 25- to 50-mg ascorbic acid per 40-mL sample should neutralize the residual chlorine that may be present in treated water. Provide a spatula and reagent grade ascorbic acid in a small vial for field preservation. The ascorbic acid should not be added to the HCL preservative in the laboratory. It can be added only in the field immediately prior to sample collection. Sodium thiosulfate is not recommended for quenching residual chlorine since it produces sulfur that can contaminate the instrument. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in separate coolers designated for storing VOC aqueous samples.

6.5 VOA vials for headspace analysis of solid samples are described in SW-846 Method 5021. Standard 125-ml wide mouth glass containers may be used for SW-846 Methods 5031 and 5032. However, the sampling procedures described in Method 5035A may minimize sample preparation analyte loss better than the procedures described in Methods 5031 and 5032. No AML SOPs based on 5031 and 5032 are currently available due to lack of commercial demand for these procedures. The original EPA methods should be used for implementation as the need occurs. These samples should be stored in separate coolers designated for VOC samples.

6.6 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Analytical data generated outside of the specified holding times should be considered to be minimum values only. Such data may be used to demonstrate that a waste is hazardous where it shows the concentration of

a constituent to be above the regulatory threshold but cannot be used to demonstrate that a waste is not hazardous.

7.0 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control contain a number of regulated compounds at high concentrations. The toxicity of these compounds present in such complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of VOCs and other organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Sample vials, which are known to contain high concentrations of vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to organic vapors.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample methanol extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Aqueous and soil samples that have undergone the purge and trap procedures should be free of volatile contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters.

9.0 PROCEDURE

9.1 Initial Demonstration of Performance - It is assumed that the method has been implemented in the laboratory so that analysis on client samples may be performed using procedures described in the remainder of this section.

9.1.1 The calibration procedure employs SW-846 Method 8260B, which recommends three internal standards and four surrogates. The EPA SW-846 procedure meets and exceeds the EPA 524.2 procedure that recommends one internal standard (Fluorobenzene) and two surrogates (BFB and 1,2-Dichlorobenzene-d_4) for calibration. See Table-1 for a more details on the internal standards (Fluorobenzene, Chlorobenzene-d_5, and 1,4-Dichlorobenzene-d_4) used in this SOP. See Table-1 and Table 5 for details on the surrogates (Dibromofluorobenzene, 1,2-Dichloroethane-d_4, 4-Bromofluorobenzene, and Toluene-d_8) used in this SOP.

9.1.2 Tune the mass spectrometer in accordance with SW-846 method requirements (Table 2). The SW-846 criteria for m/z 75 (30% to 60% of mass 95) is more stringent than the EPA 524.2 method criteria (30% to 80% of mass 95).

9.1.3 Calibrate the instrument and perform MDL studies in accordance with applicable AML standard operating procedures. Calculate method quantitation limits (MQLs), lowest level of reporting (LLRs) and reporting limits (RLs) and MDLs as described in the AML SOP. A list of MDLs, LLRs, and MQLs for various procedures is shown in Appendix 6.

9.1.4 From the spiked blank analysis data for soil and water matrices, calculate percent recoveries for laboratory control samples (LCS), LCS duplicates and their RPD as shown in Appendix 10. Statistical and administrative laboratory quality control limits for percent recoveries and RPD are also shown in Appendix 10.

9.1.5 From the spiked sample analysis data for soil and water matrices, calculate and document percent recoveries for matrix spike (MS), and MS duplicates (MSD) as shown in Appendix 11. Calculate and document RPD for the duplicate analyses as shown in Appendix 11. Statistical and administrative laboratory quality control limits for percent recoveries and RPD are also shown in Appendix 11.

9.2 Chromatographic and MS operating conditions for routine sample analysis - See Appendix 1 for a copy of the method employed for MSD-5973 and the MSD-5970.

9.3 The mass spectrometer is tuned to meet the criteria in Table 2 for the injection of 50-ng of 4-Bromofluorobenzene (1-uL injection of the BFB standard). The 50-ng calibration standard that is used in initial calibration (ICAL), initial calibration verification (ICV), and calibration verification (CCV) may also be used in the purge and trap mode to verify the tune. The mass spectrum of BFB may be acquired by taking the apex and the scans immediately preceding and following the apex. The three scans are averaged. Background subtraction is required and must be accomplished by using a single scan no more than 20 scans prior to the elution of BFB. Do not subtract part of the BFB peak. Analyses of the initial calibration cannot be performed until the criteria for 4-BFB is met. Document instrument tune in instrument printouts as shown in Appendix 2.

9.4 Purge-and-trap – A set of five (preferred number is six) ICAL calibration standards are added to the autosamplers (Archon) in positions 1 through 5. The autosampler automatically adds the ISS to each calibration point just prior to purging. Perform the purge and trap analysis procedure using instrument conditions described in Appendix 1. Aqueous samples are analyzed using the 5030C method and soil samples are analyzed using Method 5035A. When soil samples are not collected using the 5035A method, procedures based on SW-846 5030A method (NOTE: The 5030A procedure for the analysis of soil samples is no longer included in most recent SW-846 manuals) may be used to perform analysis to produce screening level data.

9.5 Tabulate the area response of the characteristic ions (Table 3) against concentration for each compound and each standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard that corresponds to each analyte is tested in TALBE 1. The RF calculation is:

$$RF = (A_s C_{is}) / (A_{is} C_s)$$

where:

A_s = Area of the characteristic ion for the compound being measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_{is} = Concentration of the specific internal standard
C_s = Concentration of the compound being measured

9.6 Calculate the mean RF (RF_m) for each compound using the five RF values calculated for each ICAL standard. Equations used are described in a number of AML SOPs.

9.7 System performance checks (SPCC) are used to evaluate instrument response and relative response factors. Five compounds are checked for minimum average response factor. These (SPCC) compounds are chloromethane; 1,1-dichloroethane; bromoform; 1,1,2,2-tetrachloroethane and chlorobenzene with the minimum relative RF being 0.10, 0.10, 0.10, 0.30 and 0.30 in order.

9.8 Calculate the standard deviation (SD) and the percent relative standard deviation (%RSD) for each target compound. Equations used are described in a number of AML SOPs including the SOP on training. The percent RSD is calculated as follows:

$$\% \text{ RSD} = ((SD)/(RF_m))*100\%$$

where:

RF_m = mean of the 5 initial RFs for a compound
SD = standard deviation for the mean of 5 RFs for the compound.

9.9 Document initial calibration in instrument printouts as shown in Appendix 2. For the ICAL to be acceptable using the response factor method, the %RSD should be less than or equal to 15% for each target compound. The % RSD for each individual Calibration Check Compound (CCC) must be less than 30%. The CCCs are 1,1-Dichloroethene; chloroform; 1,2-Dichloropropane; toluene; ethylbenzene and vinyl chloride. For drinking water samples, the %RSD should be <20% for the target compounds and surrogates (EPA 524.2 criteria).

9.10 If a % RSD greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column active sites is required before reattempting the calibration. The calibration curve must be re-analyzed if any CCC remains above 30% RSD for CCC compounds.

9.11 If a % RSD greater than 30% is measured for any target compound, alternate calibration procedures (regression methods) should be employed. Corrective action to eliminate system leak and/or column active sites is required before re-attempting initial calibration. Criteria shown in the appropriate determinative methods (e.g. 8260B, 624, and 524.2) supersede those contained in EPA SW-846 guidance documents (e.g. SW-846 Method 8000C) on analytical chromatography.

9.12 Linearity - If the % RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range and the average relative response factor may be used for quantitation. The analyst, exercising professional judgment, can use regression methods for compounds with less than 15% RSD, if decreasing or increasing trends are observed for response factors at various concentrations. Since regression methods normally weigh more toward high-level standards, weighed regression methods are recommended if the concentration of concern is near low-level standards.

9.13 If the % RSD of any detected compound is greater then 15%, use least square regression methods to calculate sample concentrations. For regression methods, the instrument response is treated as the dependent variable (y) and the standard amount is the independent variable (x). When "C_s" is used as "x", use "A_s*C_{is}/A_{is}" as "y". When "C_s/C_{is}" is used as "x", use "A_s/A_{is}" as "y". Do not include origin (0,0) as an extra calibration point.

9.14 Weighted least squares method is preferred for calculating concentrations more accurately at lower levels. A linear least squares equation ($y = a*x + b$) is preferred over non-linear calibration equations ($y = a*x^2 + b*x + c$). The coefficient of determination (COD or r^2) or correlation coefficient (r) should be used to measure the goodness of fit. For regression models to be used for quantitative purposes, the COD or r^2 must be greater than or equal to 0.99 and the correlation coefficient (r) should be greater than 0.995. A value of 1.00 for COD and/or "r" indicates a perfect fit.

9.15 Corrective action may be required if the criteria for %RSD, r, r^2, or COD are not met.

9.15.1 An initial calibration (ICAL) should be considered a single event process. Consult the QA office or check applicable EPA methods (e.g. SW-846 Method 8000C and 8260B) for additional guidance on appropriate corrective action.

9.15.2 Re-fitting of ICAL – The selected model for ICAL may be subjected to an additional re-fitting check to establish representativeness of the model for each compound in each ICAL standard. This is an optional procedure, which is recommended but not required since automated data processing procedures may not be able to accommodate it. Calculate the %D between the calculated amount (C_c) for each compound in each ICAL standard from the ICAL model and its expected value (C_e). Ideally, the %D ($(C_c-C_e) * 100 / C_e$) for each compound in ICAL standard should meet the QC criteria for %RSD. It is possible for one or more ICAL standards to have %D exceeding the %RSD criteria, while the %RSD for ICAL model is within QC limits. The re-fitting procedure may be used to identify ICAL standards that need re-analysis or dropped from the calculation of ICAL model when re-analysis was not performed.

9.15.3 Check and optimize instrument operating conditions including automated peak integration procedures for appropriateness in addressing the chromatographic needs of the specific compound with problems.

9.15.4 If the problem appears to be associated with just one of the ICAL standards, that one standard may be reanalyzed once immediately (must be within 8-hours) after the ICAL. Discard all the results from the original analysis of the standard in question and replace it with the results from re-analysis.

9.15.5 When additional calibration standards (six or seven instead of five) are analyzed, narrowing the range of concentrations to be used for determining the goodness of fit is an acceptable procedure. Five or more standards should remain for the calculation of RSD or linear regression for each compound after one or more points are removed from the ICAL. More points (6 for second order and 7 for third order) may be needed for non-linear regression. It is generally prohibited to remove data points from within a calibration range, while still retaining the extreme ends of the calibration range. Do not discard a middle point unless it appears to have an obvious problem and six or more standards were analyzed for ICAL and re-analysis of the affected standard was

not performed within the required time limits. Changes to the upper end of the calibration range will affect the need to dilute samples above the range. A change to the lower range is not recommended since it will affect the method quantitation limit (MQL) used to report data. The default MQLs, which are based on the lowest ICAL concentration are stored in the LIMS and project-specific changes are not practical.

9.15.6 The refitting procedures may lead to failed %Ds for upper level calibration standards for average response factor methods. Regression methods (linear or non-linear) are more appropriate for these compounds. Alternatively, the analyst may take labor-intensive corrective actions such as the analysis of sample extracts under dilution for the affected compounds.

9.15.7 The refitting procedures may lead to failed %Ds for lower level calibration standards for regression methods. Regression methods (linear or non-linear) are more appropriate for these compounds.

9.16 NOTE: Grand mean RSD – The procedure for the calculation of the grand mean %RSD for all target compounds in the ICAL solution when the % RSD of any detected compound is greater than 15% and the acceptability of ICAL for all the compounds if the grand mean %RSD is <15% is NOT ALLOWED.

9.17 The term "calibration verification" (CALV) is used to describe initial verification (ICV) of the initial calibration (ICAL) using second-source standards and subsequent continuing verifications (CCVs) using same-source standards. The response factors calculated during CALV are not used for sample quantitation since CALV standards are not used as "continuing calibration" standards permitted in some of the EPA methods.

9.18 Initial Calibration Verification (ICV) - Analysis of a second source ICV standard (Section 5. 13) is required before any samples can be analyzed for USACE projects. Since second source standards are not employed for the analysis of LCS/LCSD and MS/MSD samples, ICV analysis is applicable for all projects. Calculate the percent difference (%D) or %Drift for the observed response factor in ICV from the mean RF as described in Section 9.17. The %D or %drift should not exceed ±20% for each target compound. Document calibration verification in instrument printouts as shown in Appendix 2. Check the ICAL and ICV standards if it is not achievable for all the target compounds without any exception. Use professional judgment for non-USACE projects and for compounds that are not contaminants of concern (COC) for USACE projects. The ICV may be used as the beginning CCV for sample analysis.

9.19 Continuing Calibration verification (CCV) - The initial calibration curve for each compound of interest must be checked and verified once every 12-hours following ICAL and ICV analyses. This is done by analyzing a BFB tune sample and calibration verification standard (NOTE: BFB contained in the CCV standard may be used to demonstrate tune), which is usually the mid-low standard. Separate CCV analysis is not necessary for samples analyzed immediately after ICV provided the machine is demonstrated to be in tune for and/or from the ICV analysis.

9.20 Check the SPCC in the ICV and CCV standards using the same criteria listed for the initial calibration. Document calibration verification in instrument printouts as shown in Appendix 2. If the SPCC compounds for minimum response are not met, then corrective action must be taken. Corrective action usually includes cleaning the injection port, clipping the column or running methanol through the Archon system.

9.21 After checking the SPCC compounds, calculate either the percent difference (%D) (for response factors) or percent drift (%D) (for linear regression) for the CCCs using one of the following equations:

$$\%D = \%Difference = ((RF_v - RF_m) * 100)/(RF_m)$$

$$\%D = \%\ drift = ((C_v - C_m) * 100) / (C_m)$$

where:

RF_m = Average or mean response factor from ICAL
RF_v = Observed RF for the CALV (ICV or CCV) standard
C_m = True concentration of the CALV standard.
C_v = Measured concentration of the CALV standard using selected quantitation method (e.g. linear regression).

9.22 If the %D for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. For drinking water sample analyses, the %D should not exceed 30% for each target compound. If the criterion is not met (i.e., >20%), for any one of the CCCs, corrective action as described above should be taken. If the source of the problem cannot be detected, then a new five point calibration must be generated. This criterion must be met before any quantitative analysis can begin.

9.23 For specific USACE projects, client may specify that %D for the contaminants of concern (COC) should be within the CALV QC limits (<20%).

9.24 Grand mean %D – Previous versions of the EPA method(s) had provisions for the calculation of the grand mean %D for all target compounds in the CALV solution, when the %D of any detected compound is greater than 20%. The grand mean %D should be <20% for CALV to be acceptable. This procedure is discontinued. Criteria for calibration verification are compound-specific.

9.25 The internal standard responses and retention times in the check calibration standard must be evaluated immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration check (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the EICP area for any of the internal standards (in all analyses) changes by a factor of two (-50% or + 100%) from the last daily calibration check (ICV or CCV as applicable), the mass spectrometer must be inspected for malfunctions and corrections must be made as necessary. If any samples were analyzed after the system checks are invalid, those samples will need to be re-analyzed after corrections have been made.

9.26 For drinking water analyses, the absolute area of the quantitation ions of the internal standards and surrogates should be within ±30% of the area for the most recent CCV and within ±50% of the area for the same standard in ICAL.

9.27 Analysis of field samples (water) - Place the sample bottle, which has been allowed to come to ambient room temperature, into the correct position of the Archon autosampler. The Archon will automatically add the ISS to each sample analysis. Analyze samples using procedures that were employed for the analysis of the initial calibration standards.

NOTE: The process of taking a volatile aliquot destroys the validity of the sample remaining in the vial for future analysis. Multiple vials (2 to 3) vials should be collected for reanalysis of the field samples with or without dilution. When multiple vials are unavailable for sample analysis, the analyst can prepare additional aliquots for analysis by diluting 40-mL to 100-mL with reagent water in a volumetric flask and transfer the contents to two 40-mL vials immediately. This procedure involves a 2.5-fold dilution, which should be approved by the AML Project Manager for compliance with data quality objectives for the project. Additional dilutions may also be performed by the analyst at this time, if high concentrations are suspected.

9.28 Low Level Analysis of field soil samples (5035A) - Refer to AML SOP based on SW-846 Method 5035A for more details. For low level analysis, use one of the two sample vials preserved either by freezing or with sodium bisulfate solution. Place the sample bottle, which has been allowed to come to ambient room temperature, into the correct position of the Archon. The Archon will automatically add the ISS to each sample requiring analysis. Analyze the sample similarly to the initial calibration. The sample is consumed during this analysis. The analyst should use the second vial for reanalysis if QC problems were encountered and the sample contained VOCs below the upper calibration limit. Use a third vial (preserved with methanol or by freezing) to perform medium level analysis, if the low-level analysis indicated the presence of VOCs in excess of the upper calibration limit. Methanol preservation method should be employed for the third analysis, if additional sample containers are unavailable for a third low level analysis.

9.29 Medium Level Analysis of field soil samples (5035A) - Refer to AML SOP based on SW-846 Method 5035 for more details. For medium level analysis, use the sample vial that has been preserved with methanol. If freezing is employed for sample preservation, prepare a methanol extract as described in the referenced SOP. To 5-ml of water contained in a 40-mL VOA vial, add 50-uL of the methanol extract of the sample. Place the vial into the correct position of the Archon. The Archon will automatically add the ISS to each sample analysis. Analyze the sample similarly to the initial calibration. The entire sample is used by the instrument during the analysis.

9.30 Analysis of field soil samples (5030A) - This is an obsolete procedure to be used if the client fails to collect samples in accordance with the 5035 method. Open the sample bottle, which has allowed to come to ambient room temperature, and carefully weigh out approximately 5-g into a preweighed VOA vial, re-weigh the vial and record the weight. Add 5-ml of water and seal. The dilution factor is 100. The Archon will automatically add the ISS to each sample analysis. Analyze the sample similarly to the initial calibration. The entire sample is used by the instrument during the analysis.

9.31 Analysis of field waste samples (5035A) - Open the sample bottle, which has allowed to come to ambient room temperature, and carefully weigh out approximately 4-g into a preweighed VOA vial, re-weigh the vial and record the weight. Add 10-mL of methanol and sonicate for 2-minutes. To 5-mL of water, add 50-uL of the methanol-extracted sample. The dilution factor is 250. Place the water into the correct position of the Archon.

9.32 If the initial analysis of a sample target analyte exceeds the initial calibration range, the sample must be ranalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion. When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by a blank water analysis. If the blank is not free from interferences, the system must be

decontaminated. Sample analysis cannot continue until after the blank analysis demonstrates to be free from interferences.

9.33 Document sample analysis by generating instrument printouts containing quantitation reports and chromatograms as shown in Appendix 3. Generate quantitation reports and chromatograms for field samples, QC samples (blank, LCS, LCSD, MS, MSD, etc.) and standards (initial calibration, calibration verification).

9.34 Preparation and analysis of method blank (MB) samples: The MB samples for low level aqueous sample analysis are prepared in the same manner as trip blank samples by the analyst (See Section 3.4). For low-level soil samples, MB may be prepared using vials containing the sodium bisulfate preservative and the stir bar. The MB for medium level soil analyses may be prepared using quantities of methanol (50-ul) commonly used in the analysis and the stir bar. Since no differences are observed in the MB using all these three techniques, the low-level aqueous MB may be used for all three analyses. Analyze method blanks using the same procedure as associated field samples.

9.35 Preparation and analysis of matrix spike (MS) and MS duplicate (MSD) samples - The MS and MSD samples are prepared as described below:

9.35.1 The MS/MSD water samples are prepared similarly to the LCS/LCSD samples and ICAL/CCV calibration standards. Open three sample vials designated for MS and/or MSD analyses (one at a time), pour the contents into a 100-ml volumetric flask and adjust volume to the mark by removing excess sample. For low level analysis (25-ml purge), spike 15.0-ul of the 100-ug/ml working MS/MSD solution into the volumetric and mix by inverting the flask three times. This will give a final concentration of 15-ug/L. Pour the contents of the volumetric into two 40-ml VOA vials and cap them, ensuring zero headspace. Use one of them for MS and the other for MSD. Label the MS/MSD samples appropriately. When MS/MSD analyses are performed on samples containing high levels of target analytes or when additional sample vials are not collected for MS/MSD analyses, an appropriate volume of sample contained in one 40-mL vial may be diluted to 100-mL to prepare MS/MSD samples.

9.35.2 The MS/MSD soil samples for low level analysis collected using Method 5035 are prepared by manually injecting 10-ul of the 25-ug/ml working standard by puncturing the septum with a small-gauge needle. The final concentration of VOCs for a 5-g soil sample with zero % moisture will be 50-ug/kg. Employ medium level MS/MSD analyses when additional containers are not collected for low level MS/MSD analyses.

9.35.3 The MS/MSD soil samples for low level analysis collected using Method 5030A are prepared by manually weighing 5-g aliquots of the sample into two 40-ml VOA vials and fortifying them with 5-ml of organic-free water and 10-ul of the 25-ug/ml working standard. The VOA vials are capped immediately and loaded into Preceptor-II for analysis. The final concentration of VOCs for a 5-g soil sample with zero % moisture will be 50-ug/kg.

9.35.4 The MS/MSD soil samples for medium level analysis (5035 and 5030A) are prepared by fortifying 5-ml of organic-free water in two 40-ml VOA vials with 50-ul of the methanol sample extract and 10-ul of the 25-ug/ml working standard. The VOA vials are capped immediately and loaded into Preceptor-II for analysis using the soil method. The final concentration of VOCs for a sample extract prepared using 10-ml methanol and 5-g soil sample with zero %

moisture will be 10-mg/kg. Use smaller volumes of methanol extract if VOC concentrations are expected to exceed the upper limit of calibration. Use a maximum of 250-ul of methanol extract if the expected VOC concentrations are less than 1-mg/kg.

9.36 Preparation and analysis of LCS/LCSD samples: The LCS is prepared at the same concentration as the MS/MSD.

9.36.1 The water LCS/LCSD samples are prepared using the procedure described above for MS/MSD substituting organic-free water for environmental sample. The final concentration of VOCs in the LCS/LCSD samples for 25-ml purge volumes will be 15-ug/L.

9.36.2 The low level soil LCS/LCSD samples are prepared by fortifying 5-ml of sodium bisulfate solution contained in two 40-ml VOA vials with 10-ul of the 25-ug/ml working standard. The VOA vials are capped immediately and loaded into Preceptor-II for analysis using the soil method. The final concentration of VOCs for a presumed 5-g soil sample with zero % moisture will be 50-ug/kg.

9.36.3 The medium level soil LCS/LCSD samples are prepared by fortifying 5-ml of organic free water contained in two 40-ml VOA vials with 50-ul of purge and trap grade methanol and 10-ul of the 25-ug/ml working standard. The VOA vials are capped immediately and loaded into Preceptor-II for analysis using the soil method. The final concentration of VOCs for a presumed soil extract prepared from a 10-ml methanol extract of a 5-g soil sample with zero % moisture will be 10 mg/kg.

9.37 Compound Identification for Target Compounds - The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum with characteristic ions in reference mass spectrum. The reference mass spectra is established by generating the initial calibration. The characteristic ions are defined to be the three ions of the greatest relative intensity or any ions over 30% relative intensity if less then three such ions occur in the reference spectra. Compounds should be identified as present when the criteria below are met.

9.37.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

9.37.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

9.37.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

9.37.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different retention times.

9.37.5 Identification can become difficult when more than one analyte elutes chromatographically causing overlapping of spectra. It is still possible to identify a compound by closely examining the EICP of the appropriate ions. Each analyte spectrum will contain extraneous ions contributed by the co-eluting compounds.

9.37.6 Document spectral identification in instrument printouts as shown in Appendix 4 for each sample. These printouts are also required for blanks. They are not

required for spiked samples (LCS, LCSD, MS, and MSD) and calibration standards (initial and verification).

9.38 Compound Identification for non-target Compounds - A library search may be necessary when components are present that is not identified as target analytes. Guidelines for making tentative identification are:

9.38.1 Relative intensities of major ions in the reference spectrum should be present in the sample spectrum.

9.38.2 The relative intensities of the major ions should agree within ± 20%.

9.38.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

9.38.4 Ions present in the sample but not in the reference should be reviewed for possible background contamination presence of coeluting compounds.

9.38.5 Ions present in the reference but not in the sample should be reviewed for possible subtraction from sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create discrepancies.

9.38.6 Document library search results in instrument printouts as shown in Appendix 5.

10.0 CALCULATIONS

10.1 The internal standard technique is used to determine the concentration of an identified target compound. For water samples the calculation is as follows:

$$\text{Concentration (ug/L)} = \frac{(A_X)\,(Q_{IS})}{(A_{IS})\,(RF_m)\,(V_s)}$$

where:

A_X = Area of characteristic ion for compound being measured.
Q_{IS} = Quantity of internal standard injected (ng)
A_{IS} = Area of characteristic ion for the internal standard
RF_m = Mean response factor (ICAL) for compound being measured
V_s = Volume of water (ml) purged, corrected for any dilution.

10.2 For low level analysis of soil samples (e.g. sodium bisulfate preservation) use the same equation as shown above. Substitute the dry weight of soil analyzed for V_s. The dry weight of soil is determined by multiplying the wet weight (See 5035 log-in sheets) of the samples by %solids.

10.3 For medium level analysis of soil samples (methanol extracts) the calculation is as follows:

$$\text{Concentration (ug/kg)} = \frac{(A_X)\,(Q_{IS})\,(V_T)}{(A_{IS}) * (RF_m) * (W_S) * (V_A)\,(D)}$$

where:

A_X = Area of characteristic ion for compound being measured.
Q_{IS} = Quantity of internal standard injected (ng)
A_{IS} = Area of characteristic ion for the internal standard
RF_m = Mean response factor (ICAL) for compound being measured
V_T = Volume (uL) of total methanol extract
V_A = Volume (uL) of methanol extract analyzed
W_S = Wet weight (g) of sample preserved or extracted
D = % solids (divide by 100 to express it as a fraction)

10.4 Sometimes it is necessary to give an estimated quantitation value for a non-target analyte or tentatively identified compounds (TICs). Use the equations shown above for calculations, using 1.00 for the RF_m. Use total ion chromatograms for area integration of the IS and the unknown instead of characteristic ions. The result should be identified as an "estimated value" by qualifying the value with a "J" code. The report should also identify which IS was used to determine the estimated concentrations.

10.5 Percent Recovery (%R) for LCS/LCSD and, MS/MSD samples: Percent recovery (%R) is calculated as follows.

$$\%R = \frac{(x_s - x_u)}{K} x\,100$$

where:

X_s = measured value of the spiked sample

X_u = measured value of the unspiked sample

K = known amount of the spike in the sample

When calculating %R for LCS or other reference materials, X_u could be set at zero. The same procedure should be used for calculating surrogate recoveries.

10.6 Relative percent difference (RPD) for duplicates - Precision for duplicate analysis is estimated from the relative percent difference (RPD) of the concentrations (not the recoveries) measured for MS/MSD pairs, LCS/LCSD pairs or for duplicate analyses of unspiked samples. Calculate RPD as shown below:

$$RPD = \frac{|(C_1 - C_2)| * 100}{(C_1 + C_2) * (0.5)}$$

where:
C_1 = Measured concentration of the first aliquot
C_2 = Measured concentration of the second aliquot

Calculate sample concentrations for water and soil samples as described earlier.

11.0 QUALITY CONTROL

12.1 The GC/MS system must be tuned to meet BFB specifications every 12 hours.

12.2 There must be an initial calibration (ICAL) and an initial calibration verification (ICV) of the GC/MS system. During transition from a second source LCS/LCSD to a second source ICV, ICV analysis may not be necessary.

12.3 The GC/MS system must meet the CCC and SPCC criteria every 12 hours.

12.4 For water and soil samples, the laboratory has established surrogate recoveries using guidelines contained in EPA Method 8260B. The recoveries should be within the AML limits shown in Tables 5A and 5B. NOTE: USACE has established tighter performance standards for surrogate recoveries in interference-free matrices (e.g. soil and water MB, LCS, and LCSD). These limits are also used by AML for actual matrices.

12.5 A method blank must be performed on every batch of samples, every 20 samples, or every 12 hours whichever is appropriate. An instrument blank should also be performed every 24 hours or when system contamination is present.

12.6 A laboratory control sample (LCS) must be performed on every batch of samples or every 20 samples whichever is less. It is preferable to analyze the LCS in duplicate.

12.7 A matrix spike and matrix spike duplicate must be performed on every batch of samples or every 20 samples or less, whichever is appropriate. The client must be contacted if sufficient samples are unavailable to perform MS/MSD analyses. This may be an acute problem for the analysis of samples collected using Method 5035. Perform MS/MSD analyses using samples preserved in methanol if additional sample containers for low level analyses are unavailable.

13 **DATA REVIEW, VERIFICATION AND VALIDATION -** This section will provide guidance when evaluating and validating data generated by this SOP. It provides procedures to assess if data produced is valid and legally defensible. Include a checklist cover page for the raw data package and document review in a draft case narrative, while performing the review in accordance with the review checklist (See Appendix 7). Verify that sample preparation logs are complete and the instrument run logs are available for review. Anomalies and exceptions should be documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on these checklists. USACE projects may have special requirements that will require extra attention. A simple, universal procedure for evaluating choosing appropriate corrective action doe not exist to deal with situations when one or more QC parameters are outside the acceptance window. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints.

13.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) projects may have additional requirements that are related to USACE laboratory validation (CX), Department of Defense Quality Systems Manual (DoD QSM), USACE District requirements, government contract requirements, specific USACE project requirements or USACE Architect/Engineer (A/E) requirements. USACE-CX requirements (Summary of Method Quality Objectives for Method 8260B), DoD QSM requirements, and AFCEE requirements are included in Appendix 8.

13.1.1 The USACE requirements shown in Appendix 8 and elsewhere in this SOP are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may be unattainable for routine sample analysis. Since USACE MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. The laboratory may employ default QC limits based on a combination of DoD QSM limits, USACE Shell limits, laboratory statistical limits, or other administrative limits. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

13.1.2 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality objectives and/or other QC requirements. Exceptions and data qualifications necessary should be documented on the checklists and eventually on case narratives.

13.1.3 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist (Appendix 8), perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete. For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete. For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director will review the checklists (initial/date as necessary), prepare case narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

13.1.4 The blank criteria shown in Appendix 8 has been modified by USACE auditor(s) as one half of the applicable MQL (personal communications, USACE auditor, 1999).

13.2HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis and/or sample preparation.

13.2.1 WATER - The holding times for volatiles that are unpreserved are 7 days from sampling. If a preservative is used (HCl) then a 14 day holding time is applicable.

13.2.2 SOIL – A 14 day holding is used for soil and waste samples for both preparation and analysis. Special rules may apply to samples collected using

SW-846 5035A methods. See AML SOP for 5035 soil sample collection and check with the Team Leader for details.

13.2.3 TCLP – Acid-preserved TCLP samples must be analyzed within 14 days after the TCLP zero head space extraction. Method 1311 allows for up to 14 days for TCLP preparation. Total elapsed time from sample collection to TCLP-VOC determination should not exceed 28-days.

13.2.4 Holding times are established by comparing the sampling date recorded in the chain of custody documents with the date of the injection (purge and trap/GC-MS analysis). TCLP holding times must be established by comparing first the COC to the leaching date and then the leaching date to the injection date.

13.2.5 ACTION – If holding times have been exceeded, record this information in the case narrative. The user of the data may code positive results as estimated (J) for minor violation. If the holding times are greatly exceeded, the reviewer may use professional judgement to code the data including coding them as rejected (R) or invalid (I).

13.3 INSTRUMENT TUNING – Tuning and performance criteria for mass resolution, identification and sensitivity should always meet all criteria established in TABLE 2. Tuning should be checked every 12-hours and the 12-hour analytical shift begins with the injection of the tuning standard. Since the tuning compound is contained in the ICV or CCV mixture, the 12-hour analytical shift begins either with the ICV or CCV.

13.3.1 Compare mass calibration from tabular report in the raw data with each criteria form.

13.3.2 Check to see if the BFB report is present for each 12 hour period of samples analyzed.

13.3.3 Verify that the spectra is present.

13.3.4 Calculate % abundance of m/z 96/95 as a spot check.

13.3.5 ACTION – If mass calibration is outside criteria, recalibrate and rerun 4-BFB for an acceptable tune report. Reanalyze all associated samples from failed tune. If mass calibration is outside criteria, associated data may be qualified as invalid (I) by the user. For rapid turnaround projects requiring immediate response from the laboratory, the user may decide to use laboratory data as screening data since they do not meet this requirement. Check with the client representative before releasing data that do not meet tune requirements. Document QC outliers in the checklist and in the case narrative. Report detects and non-detects without qualifiers in laboratory reports. See Appendix 2 for tune documentation and Appendix 7 for documenting this review.

13.4 INITIAL CALIBRATION – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the mass spectrometer is capable of producing data within a range that is linear.

13.4.1 SPCC – Check to see that the (SPCC) compounds are within acceptable criteria which are : chloromethane; 1,1-dichloroethane; bromoform; 1,1,2,2-

tetrachloroethane and chlorobenzene with the minimum relative RF being 0.10, 0.10, 0.10, 0.30 and 0.30 in order.

13.4.2 CCC – Check to see that the CCC compounds are within acceptable criteria which are : the % RSD for each individual Calibration Check Compound (CCC) must be less than 30%. The CCCs are 1,1-Dichloroethene; chloroform; 1,2-Dichloropropane; toluene; ethylbenzene and vinyl chloride.

13.4.3 Target Compounds - Check to see that the %RSD for each target compound is within acceptable QC limit (<15%). The Method 8260B criteria for %RSD (<15%) supersede the Method 8000C criteria (<20% for GC and HPLC methods). Check to see that COD, r^2 or r are >0.99. Use procedures shown in Section 9 for a detailed description of the ICAL requirements.

13.4.4 USACE Shell criteria for target compounds - Check to see that the %RSD for each target compound is within maximum limits (<30%).

13.4.5 ACTION - If any SPCC compound does not meet the minimum criteria as stated in 11.3.1, then correct the underlying problem and re-analyze the ICAL. The calibration is considered invalid.

13.4.6 ACTION – If any CCC compound does not meet the minimum criteria as stated in 11.3.2, then correct the underlying problem and re-analyze the ICAL. The calibration is considered invalid.

13.4.7 ACTION - Target compounds – Check to see if target compounds that are contaminants of concern (COC) are outside the criteria of 15% or less for %RSD. If any of the target compounds are outside the 15% criteria, check to see if a least square linear regression equation may be used with passing QC criteria (>0.99) for the fit (COD, r^2 or r). See Appendices 2 and 3 for required calibration documentation and Appendix 7 for documenting this review.

13.4.8 ACTION – Document QC outliers on the checklist and/or the case narrative to be provided to the client. Positive results for compounds not meeting criteria may be qualified as estimated (J) by the user. Report detects and non-detects without qualifiers in laboratory reports.

13.5 INITIAL CALIBRATION VERIFICATION (ICV) – Calibration verification includes initial calibration verification (ICV) and continuing calibration verification (CCV). A second source ICV is used to check the integrity of the ICAL standards used for calibrating the instrument. The 12-hour analytical shift begins with the injection of the tuning standard (BFB) if a separate standard is used instead of including the BFB in ICV. For samples analyzed immediately following the ICAL, the ICV is used to check for internal standard areas and retention times.

13.5.1 Target Compounds - Check to see if %D for any of the target compounds is outside the QC criteria (<±20%).

13.5.2 ACTION - Check the ICAL and ICV standards for integrity. Replace one or more of the working standards (ICV first and ICAL later) for trouble shooting. Check for blank contamination for common laboratory contaminants (e.g., acetone and methylene chloride). Correct the problem(s) and repeat ICV analysis. Use professional judgment for non-USACE projects.

13.5.3 SPCC – Check to see that the (SPCC) compounds are within acceptable criteria: chloromethane; 1,1-dichloroethane; bromoform; 1,1,2,2-

tetrachloroethane and chlorobenzene with the minimum relative RF being 0.10, 0.10, 0.10, 0.30 and 0.30 in order.

13.5.4 CCC – Check to see that the CCC compounds are within acceptable criteria: the % D for each individual Calibration Check Compound (CCC) must be less than 20%. The CCCs are 1,1-Dichloroethene; chloroform; 1,2-Dichloropropane; toluene; ethylbenzene and vinyl chloride.

13.5.5 ACTION - If any SPCC compound does not meet the minimum criteria as stated in 11.4.1, then correct the underlying problem and re-analyze the CCV. If the CCV passes, the ICAL is considered valid. Otherwise, the initial calibration (ICAL) is considered invalid. A new ICAL should be run.

13.5.6 ACTION – If any CCC compound does not meet the minimum criteria as stated in 11.4.2, then correct the underlying problem and re-analyze the CCV. If the CCV passes, the ICAL is considered valid. Otherwise, the initial calibration (ICAL) is considered invalid. A new ICAL should be run.

13.5.7 ACTION - When ICAL and CCVs are acceptable based on SW-846 criteria, positive results and non-detects for target compounds not meeting the 20% %D may be reported without data qualifications for non-USACE projects. The anomalies should be described in the case narrative. Professional judgment should be used by the analyst to handle unique situations.

13.6 CONTINUING CALIBRATION VERIFICATION (CCV) – The calibration verification includes initial calibration verification (ICV) and continuing calibration verification (CCV). They are used to check that the instrument is still operating under the linear range of the initial calibration and has remained stable every 12 hours. The 12-hour analytical shift begins with the injection of the tuning standard (BFB) if a separate standard is used instead of including the BFB in ICV or CCV. For samples analyzed outside the period employed for generating ICAL, the CCV that starts the 12-hour clock is used to check for internal standard areas and retention times.

13.6.1 SPCC – Check to see that the (SPCC) compounds are within acceptable criteria: chloromethane; 1,1-dichloroethane; bromoform; 1,1,2,2-tetrachloroethane and chlorobenzene with the minimum relative RF being 0.10, 0.10, 0.10, 0.30 and 0.30 in order.

13.6.2 CCC – Check to see that the CCC compounds are within acceptable criteria: the % D for each individual Calibration Check Compound (CCC) must be less than 20%. The CCCs are 1,1-Dichloroethene; chloroform; 1,2-Dichloropropane; toluene; ethylbenzene and vinyl chloride.

13.6.3 ACTION - If any SPCC compound does not meet the minimum criteria as stated in 11.4.1, then correct the underlying problem and re-analyze the CCV. If the CCV passes, the ICAL is considered valid. Otherwise, the initial calibration (ICAL) is considered invalid. A new ICAL should be run.

13.6.4 ACTION – If any CCC compound does not meet the minimum criteria as stated in 11.4.2, then correct the underlying problem and re-analyze the CCV. If the CCV passes, the ICAL is considered valid. Otherwise, the initial calibration (ICAL) is considered invalid. A new ICAL should be run.

13.6.5 Target compounds – Check to see if %D for any of the target compounds are outside the QC criteria (<20%).

13.6.6 ACTION – For rapid turnaround projects, positive results for compounds not meeting the criteria for the CCC and SPCC may be reported in exceptional situations as screening data with data qualifications as estimated (J).

13.6.7 ACTION - When an acceptable ICAL is verified with a valid CALV, associated sample data are acceptable. Positive results and non-detects for target compounds not meeting the 20% %D requirement for CALV should not be qualified by the laboratory. The compound-specific anomaly should be described in the case narrative.

13.7 METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Sporadic cases of contamination may be difficult to control. However, daily contamination would not be acceptable. The concentration of target analytes should not exceed QC criteria (e.g. one half of the applicable PQL for USACE projects). If they do, steps shall be taken to find/reduce/eliminate the source of this contamination in the method blank with the exception of common laboratory contaminants (e.g. acetone, methylene chloride, 2-butanone or MEK, toluene). The blank criteria for these compounds equal their PQLs. Additional project-specific corrective actions may be necessary. Sample data qualification to alert blank contamination is performed by the laboratory. Sample data qualification based on all the blank results (laboratory MB, equipment blank, and other field blanks) is performed by the user (client). The processing of blanks helps eliminate reporting of false positives in project reports. To be acceptable, blank results should not exceed blank criteria. Since uniform criteria for an acceptable MB is unavailable, the laboratory will use the USACE criteria. Document this review in checklists (Appendix 7) and in case narratives.

13.7.1 Method blanks should be free of all target anaytes and other interfering non-target analytes. To be acceptable, blank results should not exceed ½ the PQL for all compounds with the exception of common laboratory (<PQL) and/or field contaminants such as acetone, methylene chloride, 2-butanone, toluene, etc..

13.7.2 Review the results of all associated blanks and verify that the method blank has been reported per matrix for each system used to analyze the associated samples. If levels greater than ½ PQL are present, inspect and correct the problem. Reanalyze all associated samples. The only exception may be quick turn around projects and client projects with criteria that may be different.

13.7.3 ACTION – Report detects with "B" qualifiers in laboratory reports for contaminants that have been identified in the associated method blank at levels greater than MDL or method reporting limit, whichever is used for reporting sample data for the project.

13.7.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, then no action is taken. Discuss the situation in the case narrative.

13.7.5 ACTION – Any compound detected in the sample which is also detected in associated method blank at levels greater than one half the PQL (equals PQL for common lab contaminants) correct the problem by re-extracting associated

samples unless other client criteria are applicable. Exceptions may include quick turn around projects. Contact client.

13.7.6 ACTION – If the sample amount is more than 10-times the blank amount, re-extraction and re-analysis of the affected sample may not be necessary if the contamination of the MB is associated with the processing of grossly contaminated samples.

13.7.7 ACTION - If the sample amounts are less than 10-times the blank level, reanalyze the method blank, and any samples containing the same contaminant. If the contamination remains, the contaminated samples of the batch should be re-prepared and reanalyzed with a new method blank and batch specific QC samples.

13.7.8 ACTION – If contaminants resulting in GC/MS peak saturation are present, all affected compounds are qualified as invalid (I). Reanalysis will be necessary.

13.7.9 ACTION – Any compound detected in the sample which was also detected in any associated blank may be qualified by the data user (not by the laboratory) as not detected ("U" code). The data user may employ this data qualification procedure when the sample concentration is less than five times the blank concentration, except for common laboratory contaminant compounds when the 10 times rule is used. This data qualification may also be performed by the laboratory if instructed to do so by the user.

13.8 SURROGATE RECOVERIES – Surrogates or system monitoring compounds are different structurally, but similar chemically to classes of target compounds. These surrogate compounds are added just prior to extraction (or purging for VOCs analysis) of the samples and the percent recoveries of the surrogates are calculated and evaluated. A surrogate is evaluated by comparing its recovery (%R) in each sample to the laboratory QC limits, project-specific DQOs, client specifications, or other criteria (e.g. EPA limits).

13.8.1 Surrogate failures in method blanks or laboratory control samples are indicative of a general method failure and should be thoroughly investigated. Samples that appear to be heavily contaminated either with target or non-target analytes may require method modification (such as the medium level method instead of low level method) even for an initial analysis. Such requests may either originate from the analyst or the client project chemist. Other consequences such as higher detection limits for the medium level analysis, additional cost for low-level re-analysis, if medium level analysis is unacceptable, etc. should also be considered in this instance. Surrogate recoveries that fail to meet appropriate acceptance criteria would indicate that a potential matrix effect is present. For USACE projects, tighter limits may apply to interference-free samples such as MB, LCS and LCSD.

13.8.2 CHECK - Verify surrogate recoveries are within QC limits for the environmental samples including MS and MSD (maximum range is 70% to 130% for USACE projects) and interference-free samples including MB, LCS and LCSD (80% to 120% for water and 75% to 125% for soil). Flag outliers with an asterisk (*). Laboratory (AML) surrogate recovery limits (80% to 120% for water and 75% to 125% for soil) are documented in Tables 5-A and 5-B). These matrix-dependent limits should be used for all samples including

associated matrix-free samples such as MB, LCS and LCSD. Document review in checklists (Appendix 7) and in case narratives.

13.8.3 ACTION – When one or more surrogate recoveries are outside control limits, the sample must be re-analyzed. Reanalysis may be performed under dilution if original analysis should indicate high levels of target and/or non-target analytes or interferences to surrogate compounds.

13.8.4 ACTION – If surrogate recoveries are again outside of the control limits on the re-analysis, then the analysis is complete and is noted in the case narrative. Both results are reported.

13.8.5 The user may have two or more data sets that may not meet surrogate recovery criteria. The analyst may mark the preferred data set on the report. The user may exercise professional judgment in selecting the appropriate data set and qualify associated data for selected compounds as estimated ("J" flag).

13.9 INTERNAL STANDARD PERFORMANCE – Internal Standard (IS) performance criteria based on GC/MS areas and retention times (RT) ensure that the GC/MS sensitivity and response are stable during each run. Document IS areas and RTs as shown in Appendix 9.

13.9.1 IS area counts must not vary by more than a factor of 2 from the associated daily calibration standard. The internal standard areas for the CCV should not differ by a factor of 2 from the initial calibration of the same standard.

13.9.2 The retention time of the internal standard must not vary by more than 30 seconds from the associated daily calibration standard.

13.9.3 ACTION – If an internal standard is outside of QC limits of the associated daily standard, then re-analysis is required with or without depending on the type of interferences that may occur from target and non-target analytes at concentrations that may be high relative to the internal standard compounds. Do not dilute the sample to the point where all target analytes are non-detect. Professional judgement must be used when dilutions are required for IS criteria.

13.9.4 ACTION – If an internal standard is outside of QC limits of the associated daily standard for the re-analysis use professional judgement to report data for the sample. It may not be advisable to dilute the sample further, which may dilute out any target analytes. Document observations on the checklist and on the case narrative submitted with client reports. Report positive sample results for compounds quantitated using the IS with QC problems as estimated with a "J" qualifier. Non-detects for the associated IS are reported with the standard "U" qualifier. If very low IS areas are obtained, indicating loss of sensitivity and/or severe interference, non-detects may need to be reported as invalid (I). Document observations in the case narrative.

13.9.5 ACTION – If an IS retention time varies by more than +/- 30 seconds, the total ion profile must be checked for any false positives or negatives. If a large shift has occurred, then professional judgement must be used as to whether reanalyze or not.

13.10 LABORATORY CONTROL SAMPLE (LCS) – The analysis of LCS samples is used to evaluate method competence in the absence of matrix effects. The LCS is evaluated by comparing the percent recovery for all of the target analytes to the laboratory QC limits, which are identical to the 3-sigma DoD QSM QC limits (Tables 7 to 10 and also in Appendix 8). See Appendix 10 for guidance on documenting LCS and LCSD concentrations and %R in QC summary report forms. The DoD QSM specifies the number of compounds that can exceed the 3-sigma QC limits based on statistical considerations (See notes under tables 7 to 10). The LCS recovery for such compounds should not exceed the marginal exceedance 4-sigma (ME) limits. Evaluation procedures may include comparison of LCS recoveries with those determined by project-specific DQOs (See project QAPP, if available), laboratory statistical control limits, or USEPA guidance. Initially, the effect of QC failure on the samples should be evaluated. Regardless of this assessment, steps shall be taken to find the source of the problem and correct it.

13.10.1 CHECK - Compare the %R results for each compound in LCS with laboratory QC limits to see if all the results are within the acceptable range. Flag outliers with "ME" if it is within the 3-sigma limits. Flag it with "ME*" if it is outside the 4-sigma ME limits.

13.10.2 CHECK - Compare observed %R results for each compound with the laboratory limits for LCS/LCSD recoveries (e.g. USACE criteria). Perform manual calculation for %R, if necessary for QC outliers. Document the number of compounds that exceeded the QC limit and compare it against the maximum recommended by DoD QSM. Document this review in checklists (Appendix 7) and in case narratives.

13.10.3 ACTION - Typically, the LCS would be reanalyzed for the failed analytes (ME*) only. If the second analysis fails, then the LCS, method blank, and all associated samples of the batch would be reanalyzed for the failed analytes only. If sufficient sample is not available for reanalysis, or if the corrective action is ineffective, discuss the situation with the client project manager (or chemist). Include a discussion of the situation in the case narrative. The case narrative should discuss the corrective action taken and any other information.

13.10.4 ACTION - For non-USACE projects, no action may need to be taken on LCS and/or LCSD recovery data alone either to reanalyze samples and/or to qualify associated samples. Providing data of known quality is sufficient for most projects. If significant deviations (e.g. number of target compound exceeding QC limits for recoveries is greater than the allowable maximum) are noticed, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements, which may be compound specific. Check with the project manager about such requirements. Professional judgment may be used by the user (client) in evaluating sample data qualification options in conjunction with other QC data for the project

13.11 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike recovery data are used to evaluate the accuracy of the analytical method on project samples. The evaluation is performed by comparing the %R for target analytes to the DoD QSM limits

established for the LCS samples. See Tables 7 to 10 and Appendix 11 for guidance on documenting MS and MSD concentrations and percent recoveries in QC summary report forms.

13.11.1 MS data evaluation is more complex than method blank or LCS data evaluation since they are complicated by matrix effects in addition to sample preparation and analysis errors. The heterogeneity of grab soil samples, and potential non-representativeness of sequentially collected water samples, etc. further complicate the evaluation since it is assumed that the native concentrations are constant in triplicate analyses. In addition, concentrations of the target analytes in the sample can also far exceed spike amounts. If the native concentration of target analytes in the sample chosen for spiking is high relative to the spiking concentration, the differences in the native concentration between the unspiked sample and the spiked samples may also become very significant.

13.11.2 In addition to their reluctance to pay for MS and/or MSD analyses, most clients may not know what samples to collect for MS analysis. Clients may not collect sufficient samples, have no knowledge of the native analyte concentrations in the chosen sample, and may not have knowledge of MS spiking levels that are appropriate either for the sample or their project. For procedures with multiple target analytes, spiking levels may be inappropriate for one or more analytes, if samples that contain target analytes are chosen either in the field or by the laboratory. If no project samples are collected for MS analysis, samples from another project used for MS analysis may not be representative of the project samples. When project samples (e.g. surface soil) are chosen, their MS recovery characteristics may be different from those of other samples (e.g. sub-surface soil) in the same batch.

13.11.3 Document MS/MSD recoveries in instrument printouts and in LIMS generated reports as shown in Appendix 11. Flag QC outliers with “ME” and “ME*” on client reports.

13.11.4 CHECK - Compare %R results for each compound with the laboratory limits for MS/MSD recoveries as described for LCS. Perform manual calculation for %R using raw data and extraction sheet information to check outliers. Document this review in checklists (Appendix 7) and in case narratives.

13.11.5 ACTION – The results of the MS are evaluated, in conjunction with other QC information, to determine the effect of the matrix on the bias of the analysis. For DoD/USACE projects, acceptance criteria may allow for marginal exceedances as described for LCS (See above). Although these criteria are not applicable to LCS data, steps should be taken to find the cause of failure and corrective action to remedy it. Matrix spike recoveries that fail to meet the appropriate acceptance criteria may indicate that a potential matrix effect is present. A review of the MSD result, if available, may confirm the matrix effect, if it is in the same direction and same order of magnitude. Check the LCS and/or LCSD recoveries for affected compounds to rule out laboratory problems.

13.11.6 ACTION - If the sample chosen for MS/MSD is inappropriate, notify the client. When additional payments are authorized for re-analysis, re-extract

and/or reanalyze the MS/MSD samples after employing cleanup procedures (if applicable), dilution techniques to minimize matrix interference, or using other project samples that appear to be clean, when sufficient quantities are available. Re-spike the sample at a higher level (e.g., at approximately two to four times the sample concentration), then reanalyze the sample based on project-specific requirements. It is the responsibility of the client at all times to select appropriate samples for MS analysis, make payments for additional analyses, and to offer necessary guidance as needed to meet their data quality objectives.

13.11.7 ACTION - If the matrix effect cannot be resolved, discuss the situation in the case narrative. No action may be taken by the laboratory to qualify associated sample data based on MS/MSD recovery data alone. Professional judgment may be used by the user (client) in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project. Document QC outliers on the checklist, client reports (*) and draw attention to the QC outliers on the case narrative.

13.12 TARGET COMPOUND IDENTIFICATION AND REPORTING – Criteria for GC/MS qualitative analysis is to minimize the number of erroneous positive or negative identifications of compounds. Document sample analyses by generating instrument printouts as shown in Appendix 3 and 4. Document review in the checklist as shown in Appendix 7. Compare observed results for each sample with adjusted MDLs, MQLs and associated method blank results (See Tables 7 to 10) to determine appropriate data qualification on sample reports (Form-1 equivalents) as shown in Appendix 12. Prepare a QC Association form (Form-4 equivalent) for samples (includes MS and MSD samples) that are associated with batch QC (MB, LCS, and/or LCSD) as shown in Appendix 12.

13.12.1 Positive compound must be within +/- 0.06 relative time (RRT) units of the standard RRT.

13.12.2 Check to see if the mass spectra of the sample compound and a laboratory generated standard reference spectrum match according to the following criteria listed in Section 9.32 of this SOP. Use criteria shown in Section 9.33 for non-target compounds.

13.12.3 ACTION – Professional judgement must be used when applying the criteria for GC/MS analysis of target analytes. The ion pattern and clusters of the extracted ion profile should match fairly well, taking into account factors such as matrix ions and column bleed. Report compounds meeting criteria as positives. Report compounds not meeting qualitative identification criteria as not detected with a "U" flag.

13.12.4 Report compounds that were detected at concentrations that are less than the MDL and/or the lowest level of reporting (LLR) as not detected with a "U" flag.

13.12.5 Report compounds that were detected at concentrations that are greater than the MDL and/or the LLR but lower than the MQL as estimated with a "J" qualifier.

13.12.6 Report compounds that were detected at concentrations that are greater than the upper limit of calibration (adjusted for sample weight/volume, dilutions, and %moisture) as estimated with an "E" qualifier to indicate that calibration range has been exceeded.

13.12.7 Reanalyze samples that contain "E" flagged compound data under dilution to yield data without such flags. Provide both sets of results for use by the client.

13.12.8 For rapid turnaround non-USACE projects, report only compounds present at levels greater than the reporting limit (RL). See AML SOP on MDL for details.

14 REFERENCES

14.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8260B, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), December 1996.

14.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

14.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

14.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5000, Sample Preparation for Volatile Organic Compounds, December 1996.

14.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5021, Volatile Organic Compounds in soils and other solid matrices using equilibrium headspace analysis, December 1996.

14.6 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5030A, Purge and Trap, July 1992.

14.7 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5030B, Purge and Trap for Aqueous Samples, December 1996.

14.8 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5030C, Purge and Trap for Aqueous Samples, Revision 3, May 2003.

14.9 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5031, Volatile Nonpurgeable Water-soluble Compounds by Azeotropic Distillation, December 1996.

14.10 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5032, Volatile Organic Compounds by Vacuum Distillation, December 1996.

14.11 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5035, Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples, December 1996.

14.12 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5035A, Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples, Draft Revision 1, July 2002.

14.13 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 5041A, Analysis for Desorption of Sorbent Cartridges from Volatile Organic Sampling Train (VOST), December 1996.

14.14 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3585, Waste Dilution for Volatile Organics, December 1996.
14.15 Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, EPA, 40 CFR Part 136, Method 624: Purgeables, October 1984.
14.16 Methods for the Determination of Organic Compounds in Drinking Water, Supplement II, EPA/600/R-92/129, Method 524.2, Measurement of Purgeable Organic Compounds in Water by Capillary column Gas Chromatography / Mass Spectrometry, August 1992.
14.17 USEPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-media, Multi-concentration, OLM02.0.
14.18 USEPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-media, Multi-concentration, OLM03.0.
14.19 USEPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-media, Multi-concentration, OLM04.0.
14.20 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.
14.21 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.
14.22 AML Standard Operating Procedures, AML-5035-SA1.
14.23 AML Standard Operating Procedures, AML-5035-SC1.
14.24 AML Standard Operating Procedures, AML-5035-SP1.
14.25 AML Standard Operating Procedures, O-5035-RA.
14.26 AML Standard Operating Procedures, O-SoilVOCs-5035A-RB.
14.27 AML Standard Operating Procedures, Q-Water-RC.
14.28 AML Standard Operating Procedures, Q-WasteMgmt-RD.
14.29 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
14.30 AML Standard Operating Procedures, Q-Controlcharts-RC.

TABLE 1

GAS CHROMATOGRAPHIC RETENTION TIMES AND INTERNAL STANDARDS FOR VOLATILE ORGANIC COMPOUNDS ON DB-624 CAPILLARY COLUMN

ANANLYTE	RETENTION TIME (minutes)	INT STD
Dichlorodifluoromethane	3.24	1
Chloromethane	3.62	1
Vinyl chloride	3.56	1
Bromomethane	3.88	1
Chloroethane	3.96	1
1,1-dichloroethene	4.67	1
Acetone	4.60	1
Methylene chloride	5.04	1
Trans - 1,2-dichloroethene	5.34	1
MTBE	5.30	1
2-butanone (MEK)	6.27	1
Cis - 1,2-dichloroethene	6.31	1
1,1-dichloroethane	5.73	1
2,2-dichloropropane	6.35	1
Bromochloromethane	6.57	1
Chloroform	6.62	1
1,1,1-trichloroethane	6.93	1
1,1-dichloropropene	7.11	1
Carbon tetrachloride	7.14	1
Benzene	7.38	1
1,2-dichlorobenzene	7.31	1
Trichloroethene	8.15	1
1,2-dichloropropane	8.44	1
Dibromomethane	8.59	1
Bromodichloromethane	8.78	1
Cis-1,3-dichloropropene	9.44	1
4-methyl-2-pentanone (MIBK)	9.60	1
Toluene	10.03	1
Trans-1,3-dichloropropene	10.27	1
1,1,2-trichloroethane	10.59	1
Tetrachloroethene	10.95	2
1,3-dichloropropane	10.90	2
Dibromochloromethane	11.32	2
1,2-dibromoethane	11.56	2
2-hexanone	10.96	2
Chlorobenzene	12.47	2
1,1,1,2-tetrachloroethane	12.59	2
Ethylbenzene	12.66	2
Meta- and para- xylene	12.88	2
Ortho-xylene	13.67	2
Styrene	13.68	2
Bromoform	14.04	2
1,3,5-trimethylbenzene	15.63	3
1,1,2,2-tetrachloroethane	14.94	3
Bromobenzene	15.06	3
n-propylbenzene	15.26	3

NOTE: The retention times (RTs) data shown above are intended for illustration only. The RTs are subject to change.

TABLE 1 (continued)

GAS CHROMATOGRAPHIC RETENTION TIMES AND INTERNAL STANDARDS FOR VOLATILE ORGANIC COMPOUNDS ON DB624 CAPILLARY COLUMN

ANANLYTE	RETENTION TIME (minutes)	INT STD
2-chlorotoluene	15.46	3
Isopropylbenzene	14.40	2
4-chlorotoluene	15.68	3
Tert butyl benzene	16.34	3
1,2,4-trimethylbenzene	16.44	3
1,3-dichlorobenzene	17.07	3
Sec-butylbenzene	16.82	3
p-isopropyltoluene	17.13	3
1,4-dichlorobenzene	17.26	3
n-butylbenzene	18.04	3
1,2-dichlorobenzene	18.09	3
1,2,3-trichlorobenzene	22.87	3
Hexachlorobutadiene	22.15	3
Naphthalene	22.31	3
1,2,4-trichlorobenzene	21.75	3
INTERNAL STANDARDS		
1. Fluorobenzene	7.69	
2. Chlorobenzene-d5	12.42	
3. 1,4-dichlorobenzene-d4	17.21	
SURROGATES		
Dibromofluoromethane	6.81	1
1,2-Dichloroethane-d4	7.22	1
4-Bromofluorobenzene	14.71	2
Toluene-d8	9.91	1

NOTE: The retention times (RTs) data shown above are intended for illustration only. The RTs are subject to change.

TABLE 2

MASS INTENSITIES SPECIFICATIONS FOR BFB (TUNING COMPOUND) (4-BROMOFLUOROBENZENE)

MASS	INTENSITY REQUIRED (RELATIVE ABUNDANCE)
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

TABLE 3

CHARACTERISTIC MASSES (m/z) FOR PURGEABLE ORGANIC VOLATILES

ANALYTE	PRIMARY ION	SECONDARY ION(S)
Chloromethane	50	52
Vinyl chloride	62	64
Bromomethane	94	96
Chloroethane	64	66
1,2-dichloroethene	96	61, 63
Acetone	58	43
Methylene chloride	84	86, 49
1,1-dichloroethene	96	61, 63
2-butanone (MEK)	72	43, 72
1,1-dichloroethane	63	65, 83
2,2-dichloropropane	77	97
Bromochloromethane	128	49, 130
Chloroform	83	85
1,1,1-trichloroethane	97	99, 61
1,1-dichloropropene	75	110, 77
Carbon tetrachloride	117	119
Benzene	78	-
1,2-dichlorobenzene	62	98
Trichloroethene	95	97, 130, 132
1,2-dichloropropane	63	112
Dibromomethane	93	95, 174
Bromodichloromethane	83	85, 127
cis-1,3-dichloropropene	75	77, 39
2-hexanone	43	58, 57, 100
Toluene	92	91
Trans-1,3-dichloropropene	75	77, 39
1,1,2-trichloroethane	83	97, 85
Tetrachloroethene	164	129, 131, 166
1,3-dichloropropane	76	78
Dibromochloromethane	129	127
1,2-dibromoethane	107	109, 188
4-methyl-2-pentanone	100	43, 58, 85
Chlorobenzene	112	77, 114
1,1,1,2-tetrachloroethane	131	133, 119
Ethylbenzene	91	106
m/p xylene	91	106
o-xylene	91	106
Styrene	104	78
Bromoform	173	175, 154
1,3,5-trimethylbenzene	105	120
1,1,2,2-tetrachloroethane	83	131, 85
Bromobenzene	156	77, 158
1,2,3-trichloropropane	75	182, 145
n-propylbenzene	91	120

TABLE 3 (continued)

CHACTERISTIC MASSES (M/Z) FOR PURGEABLE ORGANIC VOLATILES

ANALYTE	PRIMARY ION	SECONDARY ION(S)
2-chlorotoluene	91	126
Isopropylbenzene	105	120
4-chlorotoluene	91	126
tert butyl benzene	119	91, 134
1,2,4-trimethylbenzene	105	120
1,3-dibhlorobenzene	146	111, 148
sec butylbenzene	105	134
p-isopropyltoluene	119	134, 91
1,4-dichlorobenzene	146	111, 148
n-butylbenzene	91	92, 134
1,2-dichlorobenzene	146	111, 148
1,2-dibromo-3-chloropropane	75	155, 157
1,2,3-trichlorobenzene	180	182, 145
Hexachlorobutadiene	225	223, 227
Naphthalene	128	-
1,2,4-trichlorobenzene	180	182, 145
INTERNAL STANDARDS		
1. Fluorobenzene	96	
3. chlorobenzene-d5	117	
4. 1,4-dichlorobenzene-d4	152	
SURROGATES		
Dibromofluoromethane	113	
1,2-Dichloroethane-d4	65	
4-BFB	95	
Toluene-d8	98	

TABLE 4

SUMMARY OF METHOD QUANTITATION LIMITS FOR VOLATILES LISTED IN TABLE 1

Medium MDL Water 5-ml purge (ug/L)	Low MDL Water 25-ml purge (ug/L)	Low MDL Soil/Sediment Preservation by Freezing $NaHSO_4$ Preservation (ug/kg)	Medium MDL Soil/Sediment Methanol Preservation (ug/kg)
10	2	10	2000

The method quantitation limit (MQL) is the lowest concentration that can be reliably determined within specified limits of precision and accuracy during routine laboratory operating conditions. The MQL is generally the lowest non-zero standard in the calibration curve with a few exceptions. The MQLs listed herein are provided for guidance and may not always be achievable. MQLs may also be adjusted for reporting data on a dry weight basis and any dilutions that may be performed due to non-target or target interference. See Tables 7 to 10 for compound specific and procedure specific MDLs and MQLs.

TABLE 5-A

SURROGATE SPIKE RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES (ACTUAL MATRICES)

Surrogate Compound	**Water** – QC limits for %R			**Soil** - QC limits for %R		
	8260B Limits	**USACE and DoD QSM** Limits	**AML or DoD QSM** Limits	**8260B** Limits	**USACE and DoD QSM** Limits	**AML** Limits
4-Bromofluorobenzene	86-115	70-130 75-120	80-120	74-121	70-130 85-120	75-125
Dibromofluoromethane	86-118	70-130 85-115	80-120	80-120	70-130 NA	75-125
Toluene-d8	88-110	70-130 85-120	80-120	81-117	70-130 85-115	75-125
1,2-Dichloroethane-d4	80-120	70-130 70-120	80-120	80-120	70-130 NA	75-125

TABLE 5-B

SURROGATE SPIKE RECOVERY LIMITS FOR INTERFERENCE-FREE MATRICES (MB, LCS AND LCSD)

Surrogate Compound	**Water** – QC limits for %R			**Soil** - QC limits for %R		
	8260B Limits	**USACE and DoD QSM** Limits	**AML** Limits	**8260B** Limits	**USACE and DoD QSM** Limits	**AML** Limits
4-Bromofluorobenzene	86-115	80-120 75-120	80-120	74-121	75-125 85-120	75-125
Dibromofluoromethane	86-118	80-120 85-115	80-120	80-120	75-125 NA	75-125
Toluene-d8	88-110	80-120 85-120	80-120	81-117	75-125 85-115	75-125
1,2-Dichloroethane-d4	80-120	80-120 70-120	80-120	80-120	75-125 NA	75-125

TABLE 6

PREPARATION OF DAILY STANDARDS FOR ANALYSIS USING THE 100-µg/mL STANDARD SOLUTION

MSD5973 - 25-mL purge Water

Amount of STD (µL)	Volume (mL) Water	Final Conc. (µg/L)
2	100	2
5	100	5
15	100	15
25	100	25
40	100	40
60	100	60

MSD5973 - 5-mL purge Water

Amount of STD (µL)	Volume (mL)	Final Conc. (µg/L)
5	50	10
25	50	50
50	50	100
75	50	150
100	50	200
150	50	300

MSD5970 5-mL purge Water

Amount of STD (µL)	Volume (mL)	Final Conc. (µg/L)
5	50	10
25	50	50
50	50	100
75	50	150
100	50	200
150	50	300

PREPARATION OF DAILY STANDARDS FOR ANALYSIS USING THE 25 µg/mL SOLUTION

MSD 5973 Soil

Amount of STD (µL)	Weight (g)	Final Conc. (µg/kg)
2	5	10
10	5	50
20	5	100
30	5	150
40	5	200
60	5	300

MSD 5970 Soil

Amount of STD (µL)	Weight (g)	Final Conc. (µg/kg)
2	5	10
10	5	50
20	5	100
30	5	150
40	5	200
60	5	300

Table 7
Aqueous Low Level Method MDLs, MQLs, and QC Limits for LCS and MS

Volatile Organic Compounds (VOCs) Analysis by GC/MS															
Analytical Management Laboratories															
Water Low Level	Preparation Method			5030C - 25 mL											
	Analytical Method			8260B											
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**			**LCS (DOD QSM) and MS QC Limits**		**Marginal Exceedance Limits (ME)**		**RPD**
Compound	Amount	µg/L	µg/L	**µg/L**	RD =B/A	Flag	**µg/L**	RQ =C/A	flag	Lower	Upper	Lower	Upper	Limit	
	µg/L	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%	
Dichlorodifluoromethane	1.0	1.22	0.13	**0.40**	3.0		2.0	5.0		30	155	10	175	30	
Chloromethane	1.0	1.60	0.12	**0.37**	4.3		2.0	5.4		40	125	25	140	30	
Vinyl Chloride	1.0	1.42	0.12	**0.37**	3.8		2.0	5.4		50	145	35	165	30	
Bromomethane	1.0	1.34	0.12	**0.38**	3.5		2.0	5.2		30	145	10	165	30	
Chloroethane	1.0	1.64	0.16	**0.50**	3.3		2.0	4.0		60	135	50	145	30	
1,1-Dichloroethene	1.0	1.30	0.12	**0.38**	3.4		2.0	5.2		70	130	55	140	30	
Acetone	1.0	1.15	0.29	**0.92**	1.3		**5.0**	5.4		40	140	20	160	30	
Carbon Disulfide	1.0	1.39	0.12	**0.37**	3.7		2.0	5.3		35	160	15	185	30	
Methylene Chloride	1.0	2.30	0.10	**0.30**	7.6	*	**5.0**	16.5		55	140	40	155	30	
trans-1,2-Dichloroethene	1.0	1.27	0.12	**0.37**	3.4		2.0	5.4		60	140	45	150	30	
Methyl-tert-butyl-ether	1.0	1.19	0.11	**0.34**	3.5		2.0	5.8		65	125	55	135	30	
1,1-Dichloroethane	1.0	1.45	0.11	**0.36**	4.1		2.0	5.6		70	135	60	145	30	
2-Butanone	1.0	1.43	0.41	**1.28**	1.1		**5.0**	3.9		30	150	10	170	30	
cis-1,2-Dichloroethene	1.0	1.17	0.08	**0.24**	4.8		2.0	8.3		70	125	60	135	30	
2,2-Dichloropropane	1.0	1.19	0.10	**0.32**	3.7		2.0	6.3		70	135	60	150	30	
Bromochloromethane	1.0	1.10	0.10	**0.32**	3.5		2.0	6.3		65	130	55	140	30	
Chloroform	1.0	1.24	0.09	**0.29**	4.2		2.0	6.9		65	135	50	150	30	
1,1,1-Trichloroethane	1.0	1.21	0.10	**0.33**	3.7		2.0	6.1		65	130	55	145	30	
1,1-Dichloropropene	1.0	0.72	0.07	**0.22**	3.3		2.0	9.1		75	130	65	140	30	
Carbon Tetrachloride	1.0	1.19	0.10	**0.32**	3.7		2.0	6.3		65	140	55	150	30	
1,2-Dichloroethane	1.0	1.12	0.37	**1.17**	1.0	*	**5.0**	4.3		70	130	60	140	30	
Benzene	1.0	1.08	0.10	**0.31**	3.5		2.0	6.4		80	120	75	130	30	
Trichloroethene	1.0	0.81	0.08	**0.24**	3.3		2.0	8.2		70	125	60	135	30	
1,2-Dichloropropane	1.0	0.95	0.10	**0.32**	2.9		2.0	6.2		75	125	65	135	30	
Dibromomethane	1.0	1.03	0.10	**0.30**	3.4		2.0	6.6		75	125	65	135	30	
Bromodichloromethane	1.0	1.05	0.09	**0.30**	3.5		2.0	6.7		75	120	70	130	30	
cis-1,3-Dichloropropene	1.0	0.55	0.06	**0.19**	2.9		2.0	10.6		70	130	60	140	30	
4-methyl-2-pentanone	1.0	0.53	0.06	**0.20**	2.6		2.0	10.0		60	135	45	145	30	
Toluene	1.0	0.95	0.06	**0.18**	5.3	*	2.0	11.1		75	120	70	130	30	
trans-1,3-Dichloropropene	1.0	0.69	0.07	**0.22**	3.2		2.0	9.2		55	140	40	155	30	
1,1,2-Trichloroethane	1.0	1.00	0.09	**0.28**	3.6		2.0	7.2		75	125	65	135	30	
1,3-Dichloropropane	1.0	0.87	0.10	**0.32**	2.7		2.0	6.3		75	125	65	135	30	
Tetrachloroethene	1.0	0.87	0.11	**0.34**	2.6		2.0	5.8		45	150	25	165	30	
2-Hexanone	1.0	0.61	0.08	**0.25**	2.4		2.0	7.9		55	130	45	140	30	
Dibromochloromethane	1.0	0.96	0.09	**0.29**	3.3		2.0	6.9		60	135	45	145	30	
1,2-Dibromoethane	1.0	0.86	0.07	**0.22**	3.9		2.0	9.1		80	120	75	125	30	
Chlorobenzene	1.0	1.09	0.10	**0.32**	3.4		2.0	6.3		80	120	75	130	30	

Table 7 (Contd.)
Aqueous Low Level Method MDLs, MQLs, and QC Limits for LCS and MS

Volatile Organic Compounds (VOCs) Analysis by GC/MS															
Analytical Management Laboratories															
Water Low Level	Preparation Method			5030C - 25 ml											
	Analytical Method			8260B											
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**			**LCS (DoD QSM) and MS QC Limits**		**Marginal Exceedance Limits (ME)**		**RPD**
Compound	Amount	µg/L	µg/L	**µg/L**	RD =B/A	Flag	**µg/L**	RQ =C/A	flag	Lower	Upper	Lower	Upper	Limit	
	µg/L	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%	
1,1,1,2-Tetrachloroethane	1.0	1.03	0.22	**0.68**	1.5		**5.0**	7.3		80	130	75	135	30	
Ethylbenzene	1.0	0.75	0.08	**0.25**	3.0		2.0	8.1		75	125	65	135	30	
m-Xylene and p-Xylene	2.0	1.44	0.18	**0.55**	2.6		2.0	3.6		75	130	65	135	30	
o-Xylene	1.0	0.52	0.06	**0.18**	2.8		2.0	10.8		80	120	75	130	30	
Styrene	1.0	0.56	0.08	**0.24**	2.4		2.0	8.4		65	135	55	145	30	
Bromoform	1.0	0.85	0.11	**0.34**	2.5		2.0	6.0		70	130	60	140	30	
Isopropylbenzene	1.0	0.51	0.07	**0.22**	2.3		2.0	9.1		75	125	65	135	30	
1,1,2,2-Tetrachloroethane	1.0	1.10	0.09	**0.30**	3.7		2.0	6.8		65	130	55	140	30	
Bromobenzene	1.0	0.88	0.10	**0.30**	2.9		2.0	6.7		75	125	70	130	30	
n-Propylbenzene	1.0	0.71	0.09	**0.27**	2.7		2.0	7.5		70	130	65	140	30	
2-Chlorotoluene	1.0	0.69	0.08	**0.24**	2.9		2.0	8.5		75	125	65	135	30	
4-Chlorotoluene	1.0	0.72	0.09	**0.30**	2.4		2.0	6.7		75	130	65	135	30	
1,3,5-Trimethylbenzene	1.0	0.64	0.09	**0.28**	2.3		2.0	7.1		75	130	65	140	30	
tert-Butylbenzene	1.0	0.57	0.06	**0.19**	3.0		2.0	10.5		70	130	60	140	30	
1,2,4-Trimethylbenzene	1.0	0.60	0.09	**0.27**	2.2		2.0	7.4		75	130	65	140	30	
Sec-Butylbenzene	1.0	0.65	0.08	**0.25**	2.6		2.0	7.8		70	125	65	135	30	
1,3-Dichlorobenzene	1.0	0.93	0.10	**0.32**	2.9		2.0	6.3		75	125	65	130	30	
1,4-Dichlorobenzene	1.0	1.13	0.09	**0.28**	4.1		2.0	7.2		75	125	65	130	30	
1,2-Dichlorobenzene	1.0	0.91	0.09	**0.30**	3.1		2.0	6.7		75	130	65	140	30	
4-Isopropyltoluene	1.0	0.53	0.08	**0.24**	2.2		2.0	8.2		70	120	60	130	30	
n-Butylbenzene	1.0	0.56	0.07	**0.22**	2.6		2.0	9.2		70	135	55	150	30	
1,2,4-Trichlorobenzene	1.0	0.86	0.08	**0.26**	3.3		2.0	7.7		65	135	55	145	30	
1,2,3-Trichlorobenzene	1.0	1.02	0.08	**0.26**	3.8		2.0	7.6		55	140	45	155	30	
Hexachlorobutadiene	1.0	0.99	0.10	**0.31**	3.2		2.0	6.4		50	140	35	160	30	
Naphthalene	1.0	0.84	0.09	**0.27**	3.1		2.0	7.4		55	140	40	150	30	
1,2,3-Trichloropropane							2.0			75	125	65	130	30	
1,2-Dibromo-3-chloropropane							2.0			50	130	35	145	30	
Trichlorofluoromethane							2.0			60	145	45	160	30	
Mean Ratio (RD or RQ):					3.2			7.2							

MDL= ST Dev * t factor (3.14 for 7 reps)

Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 5.

Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.

LCS and MS %R limits are from the DoD QSM, Revision 2 Final, June 2002.

Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).

Table 8
Aqueous Medium Level Method MDLs, MQLs, and QC Limits for LCS and MS

Volatile Organic Compounds (VOCs) Analysis by GC/MS

Analytical Management Laboratories

Matrix: Water	Preparation Method			5030C - 5 ml											
	Analytical Method			8260B											
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**			**LCS (DOD QSM) and MS QC Limits**		**Marginal Exceedance Limits (ME)**		**RPD**
Compound	Amount	µg/L	µg/L	**µg/L**	RD =B/A	Flag	**µg/L**	RQ =C/A	flag	Lower	Upper	Lower	Upper	Limit	
	µg/L	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%	
Dichlorodifluoromethane	1.0	1.17	0.18	0.55	2.13		5.0	9.1		30	155	10	175	30	
Chloromethane	1.0	1.14	0.16	0.49	2.32		5.0	10.2		40	125	25	140	30	
Vinyl Chloride	1.0	0.74	0.20	0.64	1.16		5.0	7.8		50	145	35	165	30	
Bromomethane	1.0	0.96	0.19	0.59	1.61		5.0	8.4		30	145	10	165	30	
Chloroethane	1.0	0.76	0.19	0.60	1.26		5.0	8.3		60	135	50	145	30	
1,1-Dichloroethene	1.0	0.71	0.18	0.58	1.23		5.0	8.7		70	130	55	140	30	
Acetone	1.0	2.77	0.50	1.58	1.76		5.0	3.2		40	140	20	160	30	
Carbon Disulfide	1.0	0.80	0.17	0.53	1.49		5.0	9.4		35	160	15	185	30	
Methylene Chloride	5.0	6.69	0.18	**0.55**	12.09	*	5.0	9.0		55	140	40	155	30	
trans-1,2-Dichloroethene	1.0	0.73	0.12	0.39	1.89		5.0	12.8		60	140	45	150	30	
Methyl-tert-butyl-ether	1.0	0.80	0.18	0.55	1.44		5.0	9.0		65	125	55	135	30	
1,1-Dichloroethane	1.0	0.95	0.09	0.29	3.32		5.0	17.4		70	135	60	145	30	
2-Butanone	5.0	6.13	0.22	0.68	9.01	*	5.0	7.3		30	150	10	170	30	
cis-1,2-Dichloroethene	1.0	0.77	0.10	0.33	2.36		5.0	15.3		70	125	60	135	30	
2,2-Dichloropropane	1.0	0.99	0.13	0.41	2.44		5.0	12.3		70	135	60	150	30	
Bromochloromethane	5.0	5.08	0.10	**0.31**	16.17	*	5.0	15.9		65	130	55	140	30	
Chloroform	1.0	1.05	0.07	0.23	4.55		5.0	21.6		65	135	50	150	30	
1,1,1-Trichloroethane	1.0	0.83	0.10	0.32	2.57		5.0	15.5		65	130	55	145	30	
1,1-Dichloropropene	1.0	0.43	0.08	0.26	1.67		5.0	19.2		75	130	65	140	30	
Carbon Tetrachloride	1.0	0.75	0.11	0.35	2.15		5.0	14.4		65	140	55	150	30	
1,2-Dichloroethane	1.0	1.22	0.12	0.36	3.37		5.0	13.8		70	130	60	140	30	
Benzene	1.0	0.86	0.08	0.26	3.34		5.0	19.4		80	120	75	130	30	
Trichloroethene	1.0	0.70	0.04	0.13	5.43	*	5.0	39.1		70	125	60	135	30	
1,2-Dichloropropane	1.0	0.80	0.03	0.11	7.40	*	5.0	46.3		75	125	65	135	30	
Dibromomethane	1.0	0.79	0.09	0.28	2.82		5.0	18.0		75	125	65	135	30	
Bromodichloromethane	1.0	0.94	0.06	0.18	5.27	*	5.0	27.9		75	120	70	130	30	
cis-1,3-Dichloropropene	1.0	0.37	0.04	0.12	3.15		5.0	42.3		70	130	60	140	30	
4-methyl-2-pentanone	5.0	3.57	0.14	**0.46**	7.84	*	5.0	11.0		60	135	45	145	30	
Toluene	1.0	1.08	0.27	0.85	1.27		5.0	5.9		75	120	70	130	30	
trans-1,3-Dichloropropene	1.0	0.55	0.05	0.15	3.73		5.0	33.9		55	140	40	155	30	
1,1,2-Trichloroethane	1.0	1.00	0.05	0.14	6.93	*	5.0	34.6		75	125	65	135	30	
1,3-Dichloropropane	1.0	0.79	0.09	0.28	2.79		5.0	17.7		75	125	65	135	30	
Tetrachloroethene	1.0	0.65	0.15	0.47	1.38		5.0	10.6		45	150	25	165	30	
2-Hexanone	5.0	3.89	0.20	**0.63**	6.20	*	5.0	8.0		55	130	45	140	30	
Dibromochloromethane	1.0	0.84	0.08	0.24	3.54		5.0	21.0		60	135	45	145	30	
1,2-Dibromoethane	1.0	0.72	0.14	0.43	1.70		5.0	11.7		80	120	75	125	30	
Chlorobenzene	1.0	1.15	0.06	0.18	6.40	*	5.0	27.7		80	120	75	130	30	

Table 8 (Contd.)
Aqueous Medium Level Method MDLs, MQLs, and QC Limits for LCS and MS

Volatile Organic Compounds (VOCs) Analysis by GC/MS															
Analytical Management Laboratories															
Matrix: Water	Preparation Method			5030C - 5 ml											
	Analytical Method			8260B											
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**			**LCS (DoD QSM) and MS QC Limits**		**Marginal Exceedance Limits (ME)**		**RPD**
Compound	Amount	µg/L	µg/L	**µg/L**	RD =B/A	Flag	**µg/L**	RQ =C/A	flag	Lower	Upper	Lower	Upper	Limit	
	µg/L	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%	
1,1,1,2-Tetrachloroethane	1.0	1.14	0.22	0.70	1.62		5.0	7.1		80	130	75	135	30	
Ethylbenzene	1.0	0.69	0.09	0.28	2.47		5.0	17.8		75	125	65	135	30	
m-Xylene and p-Xylene	1.0	1.02	0.21	0.65	1.58		5.0	7.7		75	130	65	135	30	
o-Xylene	1.0	0.26	0.06	0.19	1.35		5.0	25.8		80	120	75	130	30	
Styrene	1.0	0.32	0.08	0.27	1.22		5.0	18.9		65	135	55	145	30	
Bromoform	5.0	4.40	0.13	**0.42**	10.42	*	5.0	11.8		70	130	60	140	30	
Isopropylbenzene	1.0	0.32	0.08	0.25	1.28		5.0	20.3		75	125	65	135	30	
1,1,2,2-Tetrachloroethane	1.0	1.35	0.06	0.20	6.68	*	5.0	24.8		65	130	55	140	30	
Bromobenzene	1.0	0.74	0.10	0.31	2.40		5.0	16.1		75	125	70	130	30	
n-Propylbenzene	1.0	0.50	0.08	0.24	2.11		5.0	21.0		70	130	65	140	30	
2-Chlorotoluene	1.0	0.63	0.09	0.29	2.16		5.0	17.1		75	125	65	135	30	
4-Chlorotoluene	1.0	0.51	0.09	0.28	1.81		5.0	17.6		75	130	65	135	30	
1,3,5-Trimethylbenzene	1.0	0.44	0.09	0.28	1.54		5.0	17.5		75	130	65	140	30	
tert-Butylbenzene	1.0	0.39	0.12	0.37	1.07		5.0	13.6		70	130	60	140	30	
1,2,4-Trimethylbenzene	1.0	0.42	0.09	0.30	1.41		5.0	16.9		75	130	65	140	30	
sec-Butylbenzene	5.0	4.54	0.13	**0.42**	10.90	*	5.0	12.0		70	125	65	135	30	
1,3-Dichlorobenzene	1.0	0.63	0.11	0.35	1.80		5.0	14.4		75	125	65	130	30	
1,4-Dichlorobenzene	1.0	0.93	0.11	0.34	2.78		5.0	14.9		75	125	65	130	30	
1,2-Dichlorobenzene	1.0	0.75	0.11	0.34	2.21		5.0	14.7		75	130	65	140	30	
4-Isopropyltoluene	5.0	4.17	0.10	**0.31**	13.62	*	5.0	16.3		70	120	60	130	30	
n-Butylbenzene	5.0	4.21	0.09	**0.30**	14.15	*	5.0	16.8		70	135	55	150	30	
1,2,4-Trichlorobenzene	5.0	4.87	0.06	**0.20**	24.12	*	5.0	24.8		65	135	55	145	30	
1,2,3-Trichlorobenzene	5.0	5.18	0.09	**0.29**	17.86	*	5.0	17.2		55	140	45	155	30	
Hexachlorobutadiene	5.0	5.00	0.12	**0.38**	13.30	*	5.0	13.3		50	140	35	160	30	
Naphthalene	1.0	0.91	0.27	0.84	1.08		5.0	6.0		55	140	40	150	30	
1,2,3-Trichloropropane							5.0			75	125	65	130	30	
1,2-Dibromo-3-chloropropane							5.0			50	130	35	145	30	
Trichlorofluoromethane							5.0			60	145	45	160	30	
Mean Ratio (RD or RQ):					4.6			16.6							

MDL= ST Dev * t factor (3.14 for 7 reps)

Data from MDL Studies at 1-ug/L spike (3/1/01) were combined with results from 5-ug/L spike (8/28/01) studies.

Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 5.

Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.

LCS and MS %R limits are from the DoD QSM, Revision 2 Final, June 2002.

Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).

Table 9
Soil Low Level Method MDLs, MQLs, and QC Limits for LCS and MS

Volatile Organic Compounds (VOCs) Analysis by GC/MS															
Analytical Management Laboratories															
Soil Low Level	Preparation Method			5035A											
	Analytical Method			8260B											
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**			**LCS (DoD QSM) and MS QC Limits**		**Marginal Exceedance Limits (ME)**		**RPD**
Compound	Amount	µg/kg	µg/kg	**µg/kg**	RD =B/A	Flag	**µg/kg**	RQ =C/A	flag	Lower	Upper	Lower	Upper	Limit	
	µg/kg	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%	
Dichlorodifluoromethane	5.0	5.23	0.34	**1.06**	5.0		10.0	9.5		35	135	15	155	30	
Chloromethane	5.0	6.28	0.43	**1.35**	4.7		10.0	7.4		50	130	40	140	30	
Vinyl Chloride	5.0	5.65	0.37	**1.15**	4.9		10.0	8.7		60	125	45	140	30	
Bromomethane	5.0	5.29	0.13	**0.41**	12.9	*	10.0	24.4		30	160	10	180	30	
Chloroethane	5.0	5.88	0.33	**1.05**	5.6		10.0	9.6		40	155	20	175	30	
1,1-Dichloroethene	5.0	5.48	0.37	**1.17**	4.7		10.0	8.6		65	135	55	150	30	
Acetone	5.0	6.77	1.18	**3.71**	1.8		**50.0**	13.5		20	160	10	180	30	
Carbon Disulfide	5.0	5.56	0.30	**0.93**	6.0		10.0	10.7		45	160	30	180	30	
Methylene Chloride	5.0	9.72	0.44	**1.38**	7.1		10.0	7.3		55	140	40	155	30	
trans-1,2-Dichloroethene	5.0	5.43	0.26	**0.81**	6.7		10.0	12.4		65	135	55	145	30	
Methyl-tert-butyl-ether	5.0	5.56	0.53	**1.67**	3.3		10.0	6.0		65	125	55	135	30	
1,1-Dichloroethane	5.0	5.63	0.67	**2.09**	2.7		10.0	4.8		75	125	65	135	30	
2-Butanone	5.0	3.04	0.27	**0.85**	3.6		10.0	11.8		30	160	10	180	30	
cis-1,2-Dichloroethene	5.0	5.59	0.19	**0.60**	9.4		10.0	16.8		65	125	55	135	30	
2,2-Dichloropropane	5.0	5.76	0.51	**1.59**	3.6		10.0	6.3		65	135	55	145	30	
Bromochloromethane	5.0	5.35	0.69	**2.16**	2.5		10.0	4.6		70	125	60	135	30	
Chloroform	5.0	5.55	0.32	**1.02**	5.5		10.0	9.8		70	125	65	135	30	
1,1,1-Trichloroethane	5.0	5.46	0.36	**1.15**	4.8		10.0	8.7		70	135	55	145	30	
1,1-Dichloropropene	5.0	4.91	0.55	**1.73**	2.8		10.0	5.8		70	135	60	145	30	
Carbon Tetrachloride	5.0	5.18	0.39	**1.23**	4.2		10.0	8.1		65	135	55	145	30	
1,2-Dichloroethane	5.0	6.31	0.39	**1.23**	5.2		10.0	8.2		70	135	60	145	30	
Benzene	5.0	5.40	0.51	**1.59**	3.4		10.0	6.3		75	125	65	135	30	
Trichloroethene	5.0	4.59	0.73	**2.30**	2.0		10.0	4.3		75	125	70	130	30	
1,2-Dichloropropane	5.0	5.39	0.75	**2.37**	2.3		10.0	4.2		70	120	65	125	30	
Dibromomethane	5.0	5.24	0.71	**2.22**	2.4		10.0	4.5		75	130	65	135	30	
Bromodichloromethane	5.0	5.13	0.60	**1.89**	2.7		10.0	5.3		70	130	60	135	30	
cis-1,3-Dichloropropene	5.0	4.16	1.05	**3.30**	1.3		10.0	3.0		70	125	65	135	30	
4-methyl-2-pentanone	5.0	5.10	1.27	**3.98**	1.3		**50.0**	12.6		45	145	30	165	30	
Toluene	5.0	4.18	0.55	**1.72**	2.4		10.0	5.8		70	125	60	135	30	
trans-1,3-Dichloropropene	5.0	4.60	0.66	**2.07**	2.2		10.0	4.8		65	125	55	140	30	
1,1,2-Trichloroethane	5.0	5.40	0.60	**1.89**	2.9		10.0	5.3		60	125	50	140	30	
1,3-Dichloropropane	5.0	5.09	0.37	**1.16**	4.4		10.0	8.6		75	125	70	130	30	
Tetrachloroethene	5.0	4.24	0.32	**1.00**	4.2		10.0	10.0		65	140	55	150	30	
2-Hexanone	5.0	4.64	0.58	**1.81**	2.6		10.0	5.5		45	145	30	160	30	
Dibromochloromethane	5.0	5.15	0.31	**0.97**	5.3		10.0	10.3		65	130	55	140	30	
1,2-Dibromoethane	5.0	5.26	0.36	**1.14**	4.6		10.0	8.7		70	125	60	135	30	
Chlorobenzene	5.0	4.87	0.24	**0.75**	6.5		10.0	13.3		75	125	65	130	30	

Table 9 (Contd.)
Soil Low Level Method MDLs, MQLs, and QC Limits for LCS and MS

Volatile Organic Compounds (VOCs) Analysis by GC/MS															
Analytical Management Laboratories															
Soil Low Level	Preparation Method			5035A											
	Analytical Method			8260B											
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**			**LCS (DoD QSM) and MS QC Limits**		**Marginal Exceedance Limits (ME)**		**RPD**
Compound	Amount	µg/kg	µg/kg	**µg/kg**	RD =B/A	Flag	**µg/kg**	RQ =C/A	flag	Lower	Upper	Lower	Upper	Limit	
	µg/kg	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%	
1,1,1,2-Tetrachloroethane	5.0	5.41	0.22	**0.68**	7.9		10.0	14.7		75	125	65	135	30	
Ethylbenzene	5.0	4.01	0.23	**0.74**	5.4		10.0	13.6		75	125	65	135	30	
m-Xylene and p-Xylene	5.0	8.34	0.57	**1.80**	4.6		10.0	5.6		80	125	70	135	30	
o-Xylene	5.0	3.63	0.41	**1.28**	2.8		10.0	7.8		75	125	70	135	30	
Styrene	5.0	3.74	0.40	**1.24**	3.0		10.0	8.0		75	125	65	135	30	
Bromoform	5.0	4.68	0.27	**0.84**	5.6		10.0	11.9		55	135	45	150	30	
Isopropylbenzene	5.0	3.27	0.45	**1.40**	2.3		10.0	7.1		75	130	70	140	30	
1,1,2,2-Tetrachloroethane	5.0	6.26	0.22	**0.68**	9.2		10.0	14.7		55	130	40	145	30	
Bromobenzene	5.0	4.99	0.28	**0.88**	5.7		10.0	11.4		65	120	55	130	30	
n-Propylbenzene	5.0	4.04	0.35	**1.09**	3.7		10.0	9.2		65	135	50	145	30	
2-Chlorotoluene	5.0	4.00	0.36	**1.13**	3.5		10.0	8.9		70	130	60	140	30	
4-Chlorotoluene	5.0	3.59	0.35	**1.10**	3.3		10.0	9.1		75	125	65	135	30	
1,3,5-Trimethylbenzene	5.0	3.65	0.42	**1.32**	2.8		10.0	7.6		65	135	55	145	30	
tert-Butylbenzene	5.0	3.75	0.39	**1.24**	3.0		10.0	8.1		65	130	55	145	30	
1,2,4-Trimethylbenzene	5.0	3.41	0.42	**1.31**	2.6		10.0	7.7		65	135	55	145	30	
sec-Butylbenzene	5.0	3.82	0.26	**0.81**	4.7		10.0	12.3		65	130	50	145	30	
1,3-Dichlorobenzene	5.0	4.32	0.17	**0.55**	7.9		10.0	18.3		70	125	65	135	30	
1,4-Dichlorobenzene	5.0	4.88	0.18	**0.56**	8.6		10.0	17.7		70	125	65	135	30	
1,2-Dichlorobenzene	5.0	4.60	0.30	**0.94**	4.9		10.0	10.6		75	120	65	125	30	
4-Isopropyltoluene	5.0	3.05	0.32	**1.00**	3.0		10.0	10.0		75	135	65	140	30	
n-Butylbenzene	5.0	3.11	0.28	**0.89**	3.5		10.0	11.2		65	140	50	150	30	
1,2,4-Trichlorobenzene	5.0	3.67	0.37	**1.18**	3.1		10.0	8.5		65	130	55	140	30	
1,2,3-Trichlorobenzene	5.0	4.30	0.43	**1.36**	3.2		10.0	7.4		60	135	50	145	30	
Hexachlorobutadiene	5.0	4.48	0.22	**0.70**	6.4		10.0	14.2		55	140	40	155	30	
Naphthalene	5.0	5.19	0.50	**1.58**	3.3		10.0	6.3		40	125	25	140	30	
1,2,3-Trichloropropane										65	130	50	140	30	
1,2-Dibromo-3-chloropropane										40	135	25	150	30	
Trichlorofluoromethane										25	185	10	215	30	
Mean Ratio (RD or RQ):					4.4			9.3							

MDL= ST Dev * t factor (3.14 for 7 reps)

Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 10.

Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.

LCS and MS %R limits are from the DoD QSM, Revision 2 Final, June 2002.

Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).

The aqueous limits from the DoD QSM were adopted for soil matrix.

Table 10
Soil Medium Level Method MDLs, MQLs, and QC Limits for LCS and MS

Volatile Organic Compounds (VOCs) Analysis by GC/MS															
Analytical Management Laboratories															
Soil Medium Level	Preparation Method			5035/Methanol ext.											
	Analytical Method			8260B											
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**			**LCS (DoD QSM) and MS QC Limits**		**Marginal Exceedance Limits (ME)**		**RPD**
Compound	Amount	µg/kg	µg/kg	**µg/kg**	RD =B/A	Flag	**µg/kg**	RQ =C/A	flag	Lower	Upper	Lower	Upper	Limit	
	µg/kg	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%	
Dichlorodifluoromethane	200	238	22	**68**	3.5		500	7.3						30	
Chloromethane	200	251	39	**122**	2.1		500	4.1						30	
Vinyl Chloride	200	238	19	**58**	4.1		500	8.6						30	
Bromomethane	200	216	16	**50**	4.3		500	10.0						30	
Chloroethane	200	217	14	**43**	5.1		500	11.7						30	
1,1-Dichloroethene	200	199	30	**94**	2.1		500	5.3						30	
Acetone	200	221	33	**104**	2.1		500	4.8						30	
Carbon Disulfide	200	197	31	**97**	2.0		500	5.1						30	
Methylene Chloride	200	222	35	**110**	2.0		500	4.5						30	
trans-1,2-Dichloroethene	200	199	26	**80**	2.5		500	6.2						30	
Methyl-tert-butyl-ether	200	219	22	**71**	3.1		500	7.1						30	
1,1-Dichloroethane	200	199	20	**64**	3.1		500	7.8						30	
2-Butanone	200	241	41	**130**	1.9		500	3.9						30	
cis-1,2-Dichloroethene	200	204	18	**57**	3.6		500	8.8						30	
2,2-Dichloropropane	200	192	20	**62**	3.1		500	8.1						30	
Bromochloromethane	200	218	19	**60**	3.6		500	8.3						30	
Chloroform	200	210	29	**93**	2.3		500	5.4						30	
1,1,1-Trichloroethane	200	209	35	**108**	1.9		500	4.6						30	
1,1-Dichloropropene	200	213	20	**63**	3.4		500	7.9						30	
Carbon Tetrachloride	200	206	31	**98**	2.1		500	5.1						30	
1,2-Dichloroethane	200	213	13	**40**	5.4		500	12.6						30	
Benzene	200	217	10	**30**	7.2		500	16.7						30	
Trichloroethene	200	199	35	**109**	1.8		500	4.6						30	
1,2-Dichloropropane	200	218	14	**43**	5.1		500	11.7						30	
Dibromomethane	200	217	17	**53**	4.1		500	9.5						30	
Bromodichloromethane	200	216	12	**36**	5.9		500	13.8						30	
cis-1,3-Dichloropropene	200	224	21	**65**	3.4		500	7.6						30	
4-methyl-2-pentanone	200	304	101	**318**	1.0	*	**2500**	7.9						30	
Toluene	200	228	15	**46**	5.0		500	10.9						30	
trans-1,3-Dichloropropene	200	228	26	**81**	2.8		500	6.2						30	
1,1,2-Trichloroethane	200	232	28	**89**	2.6		500	5.6						30	
1,3-Dichloropropane	200	214	19	**61**	3.5		500	8.2						30	
Tetrachloroethene	200	211	21	**67**	3.2		500	7.5						30	
2-Hexanone	200	283	84	**265**	1.1		**2500**	9.4						30	
Dibromochloromethane	200	214	20	**63**	3.4		500	8.0						30	
1,2-Dibromoethane	200	219	24	**75**	2.9		500	6.7						30	
Chlorobenzene	200	217	13	**39**	5.5		500	12.7						30	

Table 10 (Contd.)
Soil Medium Level Method MDLs, MQLs, and QC Limits for LCS and MS

Volatile Organic Compounds (VOCs) Analysis by GC/MS															
Analytical Management Laboratories															
Soil Medium Level	Preparation Method			5035/Methanol ext.											
	Analytical Method			8260B											
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**			**LCS (DoD QSM) and MS QC Limits**		**Marginal Exceedance Limits (ME)**		**RPD**
Compound	Amount	µg/kg	µg/kg	**µg/kg**	RD =B/A	Flag	**µg/kg**	RQ =C/A	flag	Lower	Upper	Lower	Upper	Limit	
	µg/kg	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%	
1,1,1,2-Tetrachloroethane	200	212	14	**45**	4.8		500	11.2						30	
Ethylbenzene	200	221	13	**41**	5.3		500	12.1						30	
m-Xylene and p-Xylene	200	219	14	**43**	5.1		500	11.6						30	
o-Xylene	200	222	13	**40**	5.5		500	12.5						30	
Styrene	200	221	11	**36**	6.1		500	13.9						30	
Bromoform	200	228	28	**89**	2.6		500	5.6						30	
Isopropylbenzene	200	220	13	**42**	5.2		500	11.8						30	
1,1,2,2-Tetrachloroethane	200	230	43	**135**	1.7		500	3.7						30	
Bromobenzene	200	223	18	**57**	3.9		500	8.8						30	
n-Propylbenzene	200	222	15	**48**	4.6		500	10.4						30	
2-Chlorotoluene	200	221	14	**44**	5.0		500	11.3						30	
4-Chlorotoluene	200	220	17	**54**	4.0		500	9.2						30	
1,3,5-Trimethylbenzene	200	225	13	**40**	5.6		500	12.5						30	
tert-Butylbenzene	200	224	19	**60**	3.7		500	8.3						30	
1,2,4-Trimethylbenzene	200	223	16	**51**	4.3		500	9.7						30	
sec-Butylbenzene	200	223	15	**46**	4.8		500	10.8						30	
1,3-Dichlorobenzene	200	217	11	**35**	6.2		500	14.3						30	
1,4-Dichlorobenzene	200	215	11	**34**	6.3		500	14.7						30	
1,2-Dichlorobenzene	200	222	14	**43**	5.2		500	11.6						30	
4-Isopropyltoluene	200	221	13	**39**	5.6		500	12.7						30	
n-Butylbenzene	200	218	12	**39**	5.6		500	12.9						30	
1,2,4-Trichlorobenzene	200	220	24	**75**	2.9		500	6.7						30	
1,2,3-Trichlorobenzene	200	226	25	**79**	2.9		500	6.3						30	
Hexachlorobutadiene	200	212	8	**24**	8.9		500	21.0						30	
Naphthalene	200	246	44	**138**	1.8		500	3.6						30	
1,2,3-Trichloropropane															
1,2-Dibromo-3-chloropropane															
Trichlorofluoromethane															
Mean Ratio (RD or RQ):					3.9			9.1							

MDL= ST Dev * t factor (3.14 for 7 reps)

Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 10.

Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.

LCS and MS %R limits are from the DoD QSM, Revision 2 Final, June 2002.

Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).

The aqueous limits from the DoD QSM were adopted for soil matrix.

Table 11
Maximum Contaminant Limits (MCL) for 524.2 Drinking Water Compounds

Regulated Compound	MCL µg/L	Trihalomethanes	MCL µg/L	Unregulated Compounds
Benzene	5	Bromodichloromethane	None	Bromobenzene
Carbon tetrachloride	5	Bromoform	None	Bromochloromethane
Chlorobenzene	100	Chlorodibromomethane	None	Bromomethane
1,2-Dichlorobenzene	600	Chloroform	None	n-Butylbenzene
1,4-Dichlorobenzene	75	Trihalomethanes, total	80	sec-Butylbenzene
1,2-Dichloroethane	5			tert-Butylbenzene
1,1-Dichloroethylene	7			tert-Butylmethylether(MTBE)
Cis-Dichloroethylene	70			Chloroethane
Trans-Dichloroethylene	100			Chloromethane
Dichloromethane	5			2-Chlorotoluene
1,2-Dichloropropane	5			4-Chlorotoluene
Ethylbenzene	700			Dibromomethane
Styrene	100			1,3-Dichlorobenzene
Tetrachloroethylene (PCE)	5			Dichlorodifluoromethane
1,1,1-Trichloroethane	200			1,1-Dichloroethane
Trichloroethylene (TCE)	5			1,3-Dichloropropane
Toluene	1000			2,2-Dichloropropane
1,2,4-Trichlorobenzene	70			1,1-Dichloropropene
1,1,2-Trichloroethane	5			cis-1,3-Dichloropropene
Vinyl chloride	2			trans-1,3-Dichloropropene
Xylene, total	10,000			Fluorotrichloromethane
				Hexachlorobutadiene
				Isopropylbenzene
				4-Isopropyltoluene
				n-Propylbenzene
				1,1,1,2-Tetrachloroethane
				1,1,2,2-Tetrachloroethane
				1,2,3-Trichlorobenzene
				1,2,3-Trichloropropane
				1,2,4-Trimethylbenzene
				1,3,5-Trimethylbenzene

Table 12
List of Available Appendices
(not included in this document)

Appendix Number	Appendix Topic
1	Instrument Printouts - Chromatographic Conditions
2	Instrument Printouts -BFB Tune Summary (Form-5 equivalents) Initial Calibration (ICAL) Summary (Form-6 equivalents) Calibration Verification (ICV and CCV) Summary (Form-7 equivalents)
3	Instrument Printouts -Quantitation Reports and Sample Chromatograms
4	Instrument Printouts -Spectral Identification
5	Instrument Printouts -Library Search
6	Method Detection Limits (MDLs) Study Results Lowest Limits for Reporting (LLRs or RL - optional) and Method Quantitation Limits (MQLs)
7	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
8	USACE Method Quality Objectives - Method 8260B, EM-200-1-3, App. I Department of Defense Quality Systems Manual, Final Revision 2 AFCEE MQOs for Method 8260B
9	Internal Standard Area and RT Reports (Form-8 equivalents)
10	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
11	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
12	QC Association Forms (Form-4 equivalents) Surrogate Recovery Summaries (Form-2 equivalents) Sample Reports (Form-1 equivalents) - Soil and Water

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices. The appendices may be available in hardcopy form or as portable document electronic files (pdf).

STANDARD OPERATING PROCEDURES

No. O-BNA-SVOCs-RD

Base Neutral and Acid (BNA) Semivolatile Organic Compounds (SVOCs) by Gas Chromatography/Mass Spectrometry (GC/MS) (EPA 625 and SW-846 8270D)

October 21, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This Analytical Management Laboratories (AML) standard operating procedure (SOP) is based on EPA SW-846 Method 8270D (, EPA Method 625, and EPA contract laboratory program (CLP) methods OLM02.0, OLM03.0, and OLM04.0. Sample preparation procedures contained in this SOP are based on EPA SW-846 and other EPA methods that are referenced in this SOP. Words and acronyms such as method and SOP are used interchangeably in this document. This SOP is used to determine semivolatile organic compounds in a variety of matrices including water, air, soil and waste. Water samples will include groundwater, surface water, drinking water, and wastewater.

1.2 This SOP can be used to quantitate most neutral, acidic and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with DB5. ***Table 1*** lists the analytes or compounds and their retention times.

1.3 The practical (PQLs) or method quantitation limits (MQLs) for each individual compound is instrument dependent and highly matrix dependent. The instruments employed at AM Laboratories are standard Agilent (formerly HP) quadrupole 5973 mass selective detectors (MSD). See ***Table 1*** for MQLs that are generally achievable.

1.4 This SOP uses gas chromatography/mass spectrometry (GC/MS) procedures. This SOP is restricted to experienced analysts in the use of GC/MS theories and skilled in the interpretation of mass spectra and their use as a quantitative tool.

1.5 This SOP is intended primarily for low-level definitive laboratory analysis of aqueous and soil samples for SVOCs. Robust extraction procedures such as manual or automated liquid-liquid extraction (SW-846 Methods 3510/3520) should be used for aqueous samples. The sonication (SW-846 Method 3550) or Soxhlet (SW-846 Methods 3540/3541) methods should be employed for soil, sediment and waste samples. With appropriate modifications, microextraction procedures (SW-846 Methods 3511/3570) may be used to support rapid turnaround analysis to support environmental remediation efforts. The microextraction procedures that are described in this SOP are similar to those described in SW-846 method 8011 and EPA Method 504.1.

2.0 SUMMARY OF METHOD

2.1 This SOP provides extraction and gas chromatographic conditions for the analysis of semi-volatile compounds. Spiked samples are used to verify the chosen extraction procedure for each matrix. Samples are extracted with methylene chloride, concentrated to 1.0-mL, and analyzed by GC/MS.

3.0 INTERFERENCES

3.1 Interferences can be caused by a number of sources in the extraction and analyses stages. Quality control measures, such as method blanks, solvent blanks and matrix spikes can be used to determine the interference.

3.2 Contamination may occur when a high-concentration and a low concentration sample are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. A high concentrated sample should be followed by the analysis of solvent to check for possible cross contamination.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatographs – Two Agilent 6890 Series II GCs are used. The systems are complete with a temperature-programmable GC for splitless injection. The systems include Agilent 7683 autosamplers and other accessories, including syringes, analytical columns and gases. The column flow rate remains constant throughout temperature program operation. The capillary column is directly coupled to the source.

4.2 Gas chromatographic column - A 30 meter x 0.25-mm ID capillary column coated with DB-5 (J&W Scientific) is employed. The film thickness is 0.25 um. Other equivalent columns may be used.

4.3 Mass Spectrometers - Two Agilent 5973 Mass Selective Detectors are used. They are capable of scanning from 35 to 800 AMU every 1-second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. ***APPENDIX 1*** shows an example of a passing tune spectra of DFTPP.

4.4 GC/MS interface is directly coupled by inserting the column into the mass spectrometer.

4.5 Data system - Hewlett Packard's Enviroquant is used to allow continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the GC program. The software allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). The software also allows integrating the abundances in any EICP between specified times or scan-number limits.

4.6 Microsyringes - 10, 25, 100, 250, 500 and 1000 uLs.

4.7 Balance - Analytical, 0.0001 grams and top loading, 0.1 grams.

4.8 Disposable pipettes - Pasteur

4.9 Test tubes - 5 ml to 8 ml, with Teflon lined screw caps.

4.10 Separatory funnels - 2000 mL

4.11 Kuderna-Danish (K-D) apparatus

- 4.11.1 10- to 15-ml concentrator tubes
- 4.11.2 Evaporation flask (500-ml), attached to concentrator tubes
- 4.11.3 Three ball macro Snyder column
- 4.11.4 Clamps

4.12 Boiling Chips - solvent extracted

4.13 Heated water bath

4.14 Microsyringes - 10-, 50-, 100-, 500-, and 1000-uL.

4.15 Sonic Disrupter - Tekmar Model TM500 Sonic Disruptor with 500-watt power and pulsing capabilities. Use with ¾" horns and for guidance on use, see AML SOP on sonicator tuning and maintenance.

4.16 Tekmar Model V1A 3/4" Horn (probe) for low concentration samples and 1/8" horn for smaller sample aliquots (high concentration samples).

4.17 Glass wool

4.18 Glass beads, solvent-rinsed, bake in 400°C oven for approximately one hour

4.19 Stainless steel table spoons

4.20 IKA Automated Soxhlet Extractors – Six 4-position units equipped with heating devices, refrigerated circulating coolers, personal computer for instrument control, and micron filters to hold samples.

4.21 Soxtec Automated Soxhlet Extractors – Three 6-position units (HT-6) and two 2-position units, equipped with temperature-controlled oil bath (Soxtec). Accessories for the Soxtec equipment will include the following: Tecator bath oil (catalog number 1000-1886), contamination-free cellulose extraction thimbles (26 mm ID x 60 mm, catalog number 1522-0034), glass extraction cups (80 mL, catalog number 1000-18200, thimble adapters (catalog number 1000-14660 and Viton seals (catalog number 1000-2516).

4.22 Circulating refrigerated coolers

4.23 Mechanical shakers

4.24 40-mL VOA vials (or equivalent) for microextraction of aqueous samples (SW-846 Method 3511) for rapid turnaround analysis (in support of remediation efforts)

4.25 Polytetrafluoroethylene (PTFE) tubes (25-mL capacity) for microscale solvent extraction (SW-846 Method 3570) of soil and waste samples for rapid turnaround analysis (in support of remediation efforts)

4.26 Mechanical centrifuge

4.27 N-Evap from Organomation equipped with a tank of high purity nitrogen (Praxair High Purity Grade or equivalent) and a column of activated carbon for removing potential contaminants from the gaseous stream. CAUTION: Do not use plasticized tubing between the carbon trap and the sample, since it may introduce contaminants.

4.28 GC injection vials, with calibration marks at 0.5, 1.0, 1.5 and 2.0-mL levels. Verify volume calibration for each batch of vials as described in AML SOP on volumetric measurements. A quick check may be made by transferring 0.5-mL aliquots of methylene chloride using a calibrated 1000-µL syringe to the GC vial and checking the alignment of the volume with the mark at 0.5-, 1.0-, 1.5-, and 2.0-mL levels.

5.0 REAGENTS

5.1 It is assumed that all inorganic/organic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where available. Any other reagents must demonstrate high purity without lessening the accuracy of the determination.

5.2 Organic free reagent water - All references to water in this method will be assumed to be organic free reagent water, except when referring to field samples.

5.3 Sulfuric acid solution

5.3.1 Slowly add 50 mL of sulfuric acid to 50 mL of water (1:1 v/v)

5.4 Solvents

5.4.1 Acetone – GC grade

5.4.2 Hexane – GC grade

5.4.3 Methylene chloride - GC grade

5.5 Stock Standard solutions - These are purchased from Ultra Scientific (catalog number SVM-8270) at a concentration of 1000-ug/mL. Additional related information (surrogates, internal standards, miscellaneous compounds, etc.) is shown in ***Table 3***.

Stock standard solutions expire one year from the date of preparation. Replace sooner if signs of degradation are observed.

5.6 Semivolatiles GC/MS tuning standard solution, which contains DFTPP, 4, 4'-DDT, PCP, & Benzidine, each at a concentration of 1000-ug/mL is purchased from Ultra (cat # GCM-150). Prepare a 50-ug/ml standard by diluting 1.0-ml of the Ultra Scientific standard to 20-ml with methylene chloride. The tuning standard solution expires one year from the date of preparation. Replace sooner if signs of degradation are observed.

5.7 Working standards are prepared from the Stock Standard Solutions as described in Table 3, which includes appropriate dilutions that are needed to make each solution. The working standards are prepared at five or six concentration levels for each parameter of interest: 10-ug/mL, 20-ug/mL, 50-ug/mL, 70-ug/mL, 100-ug/mL, and 150-ug/mL. When six levels of standards are prepared, the analyst may use all six points or drop one point, if curvature is suspected. The analyst can drop either the lowest point or the highest point (depending on project requirements) but not any of the middle points. These standards are replaced every six months or sooner depending on the extent that they are used.

5.8 Surrogate spiking solution standard - The laboratory employs six surrogates (3-BN and 3-A) as shown in Table 5. The surrogate compounds are phenol-d6, 2-fluorophenol, 2, 4, 6-tribromophenol, nitrobenzene-d5, 2-fluorobiphenyl and p-terphenyl-d14. Surrogates solution standards are purchased from Ultra Scientific (Catalog number ISM-336x). Stock standard solutions expire one year from the date of preparation. Replace sooner if signs of degradation are observed. Spike 0.5-mL of the surrogate solution into each sample including all QC. The final concentration in the extract (1-mL) will be 50-µg/mL for the BN surrogates and 75-µg/mL for acid surrogates.

5.9 Matrix Spike spiking solution is prepared from the Stock Standard Solutions. Dilute 1-mL of the stock solution (1000-ug/mL) to 20-mL with acetone to prepare the matrix spike spiking solution at a concentration of 50-ug/mL. Spike 1-mL into the MS and MSD samples. These spiking solutions expire one year from the date of preparation. Replace sooner if signs of degradation are observed.

5.10 LCS spiking solution is prepared from a second source of Stock Standard Solutions. Dilute 1-mL of the stock solution (1000-ug/mL) to 20-mL with acetone to prepare the matrix spike spiking solution at a concentration of 50-ug/mL. Spike 1-mL into the LCS and LCSD samples. These spiking solutions expire one year from the date of preparation. Replace sooner if signs of degradation are observed.

5.11 Sodium Hydroxide Solution (10N) - Dissolve 40 grams of sodium hydroxide solution into 100 mLs of reagent water.

5.12 Sodium chloride (ACS Reagent grade) for microextraction of aqueous samples.

5.13 Anhydrous sodium sulfate (ACS Reagent grade), granular for drying non-aqueous samples and sample extracts. Analyze a method blank to check for interference for this analysis from the drying agent. Purify when necessary by heating at 400°C for 4 hours in a shallow tray, or by precleaning with methylene chloride. If the sodium sulfate is precleaned with methylene chloride, analyze a method blank again to check for any remaining interference from sodium sulfate.

6.0 SAMPLE PRESERVATION HANDLING & STORAGE

6.1 One liter amber glass bottles with Teflon lined screw caps may be used for collecting aqueous samples in accordance with EPA SW-846 method 8270D. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 2 bottles for each water sample to be collected. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. See project specific field sampling plan (FSP) for sample collection and preservation instructions. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.

6.2 One 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples in accordance with EPA SW-846 Method 8270D. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each solid sample. Quality control samples such as MS and MSD should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. These samples may be shared with samples collected for other analyses such as chlorinated pesticides (8081A), PCBs (8082) diesel range organics (DRO, 8015-mod.) and other pesticides (8140, etc.).

6.3 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Analytical data generated outside of the specified holding times must be considered to be minimum values only. Such data may be used to demonstrate that a waste is hazardous where it shows the concentration of a constituent to be above the regulatory threshold but cannot be used to demonstrate that a waste is not hazardous. The holding time for the extraction of aqueous samples, which includes TCLP leachates is 7 days from the date of sample collection or TCLP leachate preparation. The holding times for the extraction of soil, sediment and waste samples, which includes TCLP leachate preparation is 14 days from the date of sample collection. The holding time for the instrumental analysis of sample extracts is 40 days from the date of aqueous or solid sample extraction.

7.0 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control contain a number of regulated compounds at high concentrations. The toxicity of these compounds present in such complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Aqueous and soil samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters after the solvent(s) have been allowed to evaporate in a hood.

9.0 SAMPLE EXTRACTION PROCEDURES

9.1 Preparation of Sediment and Soil Samples

9.1.1 Sample Extraction by Sonication – SW-846 Method 3550C. See AML SOP on sonicator tuning and SW-846 Method 3550C for additional guidance.

9.1.1.1 Weight out 30-g of the soil sample to three significant numbers (to ±0.1-g) into 500-mL beakers. Record the exact weight in the organic Extraction logbook (See Appendix 13 for documentation). Add enough sodium sulfate (60-g) to dry the soil and mix well with a spoon. When the soil is dry (freely flowing powder), add 0.5-mL of surrogate solution to all the samples (including QC) and 1-ml of the appropriate spiking solutions to the LCS/LCSD & MS/MSD.

9.1.1.2 Add 100-mL of the solvent mixture (methylene chloride: acetone 50:50 v/v) to the beaker and mix the contents with a spoon for 1 minute. Place the bottom surface of the ¾" horn about ½" below the surface of the solvent, but above the sediment layer. The horn should be properly tuned in accordance with the manufacturer instructions (see AML SOP on sonicator tuning). Extract ultrasonically for 3 minutes, with output control knob set at 10 (full power) and with mode switch on Pulse (pulsing energy rather than continuous energy) and percent-duty cycle knob set at 50% (energy on 50% of time and off 50% of time). Verify that the soil is free flowing and not in clumps and that the slurry has a smooth rolling action with each pulse of energy. Decant the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus.

9.1.1.3 Extract the sample twice more with 100-mL aliquots of methylene chloride/acetone solvent mixture. The solution should be sonicated for 3 minutes each time as described in Section 9.1.1.2. Decant the solution in to the same K-D as in the previous step.

9.1.1.4 Add two boiling chips to the K-D flask and attach a three-ball Snyder column. Prewet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1- to 5-ml, remove the K-D flask and allow to cool for at least 10 minutes.

9.1.1.5 Concentrate the extract by nitrogen blow-down to 1.0-mL as described below: Place the concentrator tube in the N-Evap water bath and evaporate the solvent volume to the required level using a gentle

stream of clean, dry nitrogen. The internal walls of the tube must be rinsed down several times with the appropriate solvent during the operation. During evaporation, the solvent level in the tube must be positioned to prevent water from condensing into the sample (i.e., the solvent level should be below the level of the water bath). Under normal operating conditions, the extract must not be allowed to become dry. Please note that a few compounds (e.g. phenols, nitrobenzene, etc.) may be lost when the volume of solvent is reduced below 1-mL. Adjust the final volume to 1-mL with methylene chloride as described in Section 9.1.1.6.

9.1.1.6 Transfer the sample extract from the K-D tube to a calibrated GC vial with volume markings (Section 4.28) using additional methylene chloride as necessary for quantitative transfer. Continue the nitrogen blow-down to reduce the volume just below 1.0-mL. Adjust the volume to the 1.0-mL mark with methylene chloride using a syringe. Crimp the vial shut with a Teflon-lined septum. Proceed to analysis by GC/MS.

9.1.2 Sample Extraction by Automated Soxhlet Procedure A – SW-846 Method 3541

9.1.2.1 Automated Soxhlet Extraction Procedure-A involves IKA equipment, which are capable of handling 30-g sample aliquots. The set-up consists of six 4-position extractors to handle a total of 24 samples, which will include up to 5 QC samples (MB, LCS, LCSD, MS, and MSD). The six extractors, equipped with independent heating and cooling systems are controlled by a single personal computer. Follow manufacturer instructions for setting up and operating the IKA Soxhlet equipment.

9.1.2.2 Weight out 30-g of the soil sample to three significant numbers (±0.1-g) into 500-mL beakers. Record the exact weight in the organic Extraction logbook (See Appendix 13 for documentation). Add enough sodium sulfate (60-g) to dry the soil and mix well with a spoon. When the soil is dry (freely flowing powder), the sample is ready for Soxhlet extraction.

9.1.2.3 Clean and completely assemble the IKA equipment. Raise the condenser from the top vessel. Assemble the top vessel with the stainless steel filter base and a new disposable Teflon micron-sized filter paper. Transfer the soil/drier mixture quantitatively to the top vessel. Add 0.5-mL of surrogate solution to all the samples (including QC) and 1-ml of the appropriate spiking solutions to the LCS/LCSD & MS/MSD samples. Add 100-mL of the solvent mixture (methylene chloride: acetone 90:10) to the bottom vessel and tightly screw the top vessel on to the bottom vessel containing solvent. Replace the condenser unit on the top vessel. Turn the unit on and allow the unit to cycle (45-minutes). Completely drain the solvent from the sample chamber using the instrument cycle control mechanism. Repeat the extraction process twice using the same solvent contained in the

bottom vessel. Decant the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus.

9.1.2.4 Add two boiling chips to the K-D flask and attach a three-ball Snyder column. Pre-wet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1- to 5-ml, remove the K-D flask and allow to cool for at least 10 minutes.

9.1.2.5 Concentrate the extract by nitrogen blow-down to 1.0-mL. Proceed to analysis by GC/MS.

9.1.3 Sample Extraction by Automated Soxhlet Procedure B – SW-846 Method 3541

9.1.3.1 Automated Soxhlet Extraction Procedure-B involves Tecator Soxtec equipment, which are capable of handling only 10-g sample aliquots. The set-up consists of three 6-position extractors and two 2-position extractors to handle a total of 22 samples, which will include up to 5 QC samples (MB, LCS, LCSD, MS, and MSD). The five extractors are operated independent using three hot oil baths equipped with independent controllers. The condensers are cooled using a circulating refrigerant bath. Follow manufacturer instructions for setting up and operating the Soxtec equipment.

9.1.3.2 Weight out 10-g of the soil sample to three significant numbers (±0.1-g) into 500-mL beakers. Record the exact weight in the organic Extraction logbook (See Appendix 13 for documentation). Add enough sodium sulfate (10-g) to dry the soil and mix well with a spoon. When the soil is dry (freely flowing powder), transfer the entire contents of the beaker quantitatively to the rigid paper thimbles suspended from the stainless steel holders. Add 0.5-mL of surrogate solution to all the samples (including QC) and 1-ml of the appropriate spiking solutions to the LCS/LCSD & MS/MSD samples.

9.1.3.3 Clean and completely assemble the Soxtec extractors. Check the heating oil level in the automated Soxhlet unit and add oil if needed. See service manual for details. Set the temperature on the service unit at 80°C. Press the “MAINS” button and verify that the switch lamp is now in the “ON” position. Set the flow rate for the circulator at approximately 2-L/min to minimize solvent loss from the condensing unit.

9.1.3.4 Immediately transfer the thimbles containing the weighed samples into the condensers. Raise the knob to the "BOILING" position. The magnet will now fasten to the thimble. Lower the knob to the "RINSING" position. The thimble will now hang just below the condenser valve.

9.1.3.5 Insert the extraction cups containing boiling chips, and load each with 50-mL of extraction solvent. Using the cup holder, lower the locking

handle, ensuring that the safety catch engages. The cups are now clamped into position. (The seals must be pre-rinsed or pre-extracted with extraction solvent prior to initial use.)

9.1.3.6 Move the extraction knobs to the "BOILING" position. The thimbles are now immersed in solvent. Set the timer for 60 minutes. The condenser valves must be in the "OPEN" position. Extract for the preset time.

9.1.3.7 After rinse time has elapsed, close the condenser valves by turning each a quarter-turn, clockwise. When all but 2- to 5-mL of solvent have been collected, open the system and remove the cups.

9.1.3.8 Transfer the contents of the cups to 15-mL graduated, conical-bottom glass tubes attached to the K-D flasks. Rinse the cups using methylene chloride and add the rinsates to the glass tubes.

9.1.3.9 Add two boiling chips to the K-D flask and attach a three-ball Snyder column. Pre-wet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1- to 5-ml, remove the K-D flask and allow to cool for at least 10 minutes. Concentrate the extract by nitrogen blow-down to 1.0-mL. Proceed to analysis by GC/MS.

9.1.4 Microscale Solvent Extraction (SW-846 Method 3570) – This procedure is not a substitute for procedures that should be followed for dealing with high concentration samples as described in standard extraction methods (3550 and 3541). This procedure may be used with client approval in support of remediation efforts and for projects requiring fast turnaround times and relatively high detection limits. Project data quality objectives must be met when this procedure is used and concurrence and/or approval from clients (Check with AML Project Manager) is also required prior to using this extraction procedure.

9.1.4.1 Weight out 2.5-g (±0.5-g) of the soil sample to three significant numbers (±0.01-g) into 25-mL PTFE vials which has a PTFE screw cap. Record the exact weight in the organic Extraction logbook (See Appendix 13 for documentation). Add enough sodium sulfate (2.5-g) to dry the soil and 5 to 10 pre-cleaned glass beads for mixing the contents. Wipe the lips and threads of the tube with paper towels.

9.1.4.2 Add 0.5-mL of surrogate solution to all the samples (including QC) and 1-ml of the appropriate spiking solutions to the LCS/LCSD & MS/MSD samples. Add 12-mL of methylene chloride to the tube and cap tightly. Shake the tubes vigorously until the slurry is free flowing. Use a mechanical shaker or vortex mixer, if necessary, breaking up any chunks with a metal spatula. Add more anhydrous sodium sulfate and manually mix as necessary to produce a free flowing, finely divided slurry.

9.1.4.3 Extract the samples using a mechanical shaker or a rotating end-over-end unit for four hours. Decant or pipet the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus. Extract the soil sample in the PTFE tube twice with approximately 5-mL methylene chloride and shaking vigorously by hand for two minutes. Rinse the sodium sulfate with 2 to 3-mL of methylene chloride.

9.1.4.4 Add two boiling chips to the K-D flask and attach a three-ball Snyder column. Pre-wet the Snyder column with 1-mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1- to 5-ml, remove the K-D flask and allow to cool for at least 10 minutes.

9.1.4.5 Concentrate the extract by nitrogen blow-down to 1.0-mL. Proceed to analysis by GC/MS.

9.2 Preparation of Water Samples

9.2.1 Manual Liquid-Liquid Extraction (SW-846 Method 3510C)

9.2.1.1 Using a black marker, mark the meniscus of the water in the sample container. Pour the entire contents of the container into a 2-liter separatory funnel. Rinse the sample container with 3 aliquots of 20-mls methylene chloride and place the rinse in the separatory funnel. The volume of water contained in the sample container should be measured as describe below before the container is discarded. Add tap water carefully to the sample container up to the marked meniscus. Pour the water from the container into a 1-liter graduated cylinder. Record this measured volume in the SVOC Extraction logbook (See Appendix 13 for documentation).

9.2.1.2 An alternate procedure, which may be employed to measure out water samples, is described below. Using a 1-Liter graduated cylinder, measure out 1.00 liter ± 0.005 Liters (or lower volumes as low as 100-mL for high concentration samples) of the sample, and transfer it to a 2000-mL separatory funnel. Record the exact volume in the organic Extraction logbooks. This alternate procedure may be used to measure out lower sample volumes for the re-extraction of heavily contaminated samples with surrogate recoveries outside the QC limits, which are diluted out during analysis. This procedure may also be used to measure out smaller aliquots (e.g. 500-mL) of the sample for MS/MSD analysis, when additional sample containers were not collected for these analyses.

9.2.1.3 Using pH indicating paper (0-14 range for general purpose use and narrower range such as 0 to 4 and 10 to 14, as necessary for more accurate measurements) record the pH of the sample. This is

accomplished by dipping the tip of a Pasteur pipette into the water and placing the droplets of water on the pH paper. Never place the pH paper into the water sample. Add 0.5-mL of surrogate solution to all of the samples (including QC) and 1-ml of the appropriate spiking solution to the LCS/LCSD & MS/MSD. Adjust the pH to below 2 using the 50/50 sulfuric acid solution.

9.2.1.4 Add 60-mLs of methylene chloride to the separatory funnel and shake for 2 minutes. Take care to vent the separatory funnel to relieve excess pressure. Allow the organic layer to separate from the water layer for a minimum of 10 minutes. ***NOTE:*** *If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the solvent extract in an Erlenmeyer flask. If the emulsion cannot be broken (recovery of < 80% of the methylene chloride, corrected for the water solubility of methylene chloride), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in Method 3520, Continuous Liquid-Liquid Extraction.* Drain the extract through a funnel plugged with glass wool and covered by sodium sulfate into a K-D apparatus. Repeat the extraction 2 more times (total 3 extractions) using 60-mLs of methylene chloride. Combine the extracts into a single KD vessel.

9.2.1.5 Adjust the pH of the sample to above 12 using the 10N sodium hydroxide solution and a pH indicating paper. Add 60-mLs of methylene chloride to the separatory funnel and shake for 2 minutes. Take care to vent the separatory funnel to relieve excess pressure. Allow the organic layer to separate from the water layer for a minimum of 10 minutes. Drain the extract through a funnel plugged with glass wool and covered by acidified sodium sulfate into the same K-D apparatus as in 6.3.2. Repeat the extraction 2 more times using 60-mLs of methylene chloride. Combine the extracts into a single KD vessel.

9.2.1.6 Add two boiling chips to the K-D flask and attach a three ball Snyder column. Prewet the Snyder column with 1 mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood. When the extract reaches approximately 1.0 ml- 5.0 ml, remove the K-D flask and allow cooling for at least 10 minutes.

9.2.1.7 Concentrate the extract by nitrogen blow-down to 1.0 mLs. Proceed to analysis by GC/MS.

9.2.2 Microextraction (SW-846 Method 3511) - This procedure should be used only in support of remediation efforts and for projects requiring relatively high

detection limits. The Project Manager for specific projects may approve this extraction procedure. Samples may be collected in 40-mL VOA vials for this method if relatively volatile analytes are also included analysis using this determinative procedure. If they are collected in 1-L amber bottles, an aliquot may be transferred to a 40-mL VOA vial (including two additional aliquots for the sample selected for MS/MSD samples). Use reagent water contained in 40-mL vials for MB, LCS, and LCSD analyses. Use alternate pre-cleaned glass vials equipped with PTFE-lined caps, if the centrifuge does not accommodate 40-mL VOA vials.

9.1.2.1 Remove the cap from the VOA vial and use a disposable pipette to remove approximately 5-mL of water. Work with a single field or QC sample at a time and label the vial and the cap to minimize cross-contamination. Weigh the capped vial to the nearest 0.1-g and record it in the logbook. **NOTE:** Re-weigh the empty vial when the sample extraction procedure is completed (after completing Section 9.2.2.5) or weigh the vial before using it for microextraction. The difference between the two weights (g) should be used as sample volume (mL) assuming a density of 1.0 for aqueous samples.

9.1.2.2 Use a surrogate spiking solution (in acetone) that is 10-times more concentrated than those used for other extraction procedures (see Section 5.8) to maximize recoveries. Spike with 50-µL of the surrogate solution (10-times lower volume than for other extraction methods).

9.1.2.3 For spiked sample analysis, use LCS or MS spiking solutions (in acetone) that is 10-times more concentrated than those used for other extraction procedures (see Sections 5.9 and 5.10) to maximize recoveries. Spike with 100-µL of the LCS/MS spike solution (10-times lower volume than for other extraction methods).

9.1.2.4 Add exactly 2.0-mL (**NOTE:** This volume (2-mL) is the final extract volume for this procedure although half this volume is used for instrumental analysis) of methylene chloride using a gas-tight syringe or a class A volumetric pipette and approximately 12-g of anhydrous sodium chloride to the VOA vials. Replace the cap. Shake the vials vigorously for five minutes or until the sodium chloride dissolves completely. The agitation may be performed using a mechanical shaker, if necessary. Briefly allow the phases to settle, then centrifuge at 500 times the gravity for 15-minutes.

9.1.2.5 Tilt the VOA vial to collect the lower methylene chloride to one section of the vial bottom. Using a 1-mL gas-tight syringe, transfer approximately 1.5-mL of the lower methylene chloride layer to 1 2-mL vial with a PTFE-lined cap, carefully excluding any water. Add a small amount (approximately 50-mg) of anhydrous sodium sulfate to the vial, cap, and shake for two minutes.

9.1.2.6 Using the 1-mL gas-tight syringe, transfer exactly 1.0-mL of the dried extract to an injection vial. Proceed to analysis by GC/MS.

10 GC/MS CALIBRATION PROCEDURE

10.1 Chromatographic conditions (See also Appendix 1 for documentation):

Injection port: 250°C

Transfer line temperature: 300°C
Split ratio: 5:1
Split flow: 6-ml/min.
Carrier gas flow rate: 15 ml/min.
Initial temperature: 50°C, hold for 2-minutes
Temperature program: 6°C /min
Final temperature: 300°C, hold for 2-minutes

10.2 Initial calibration - the MS operation conditions

Mass range: 40 to 600-AMU
Scan rate: 1-sec/scan
Source temperature: 230°C

10.3 Mass Spectrometer Tune and GC Column Performance

10.3.1 The mass spectrometer is tuned (See also Appendix 1 for documentation) to meet the criteria in ***TABLE 2,*** *every 12 hours*, for the injection of 50-ng of DFTPP, 4, 4'-DDT, PCP, & Benzidine tuning solution (1-ul injection of the tuning standard). Inject the tuning standard and using the mass abundances determine if the criteria is met for DFTPP as stated in ***TABLE 2***. Background subtraction should be straight-forward only to eliminate column bleed or instrument background ions. If it is not met, retune the mass spectrometer and reanalyze the DFTPP standard. Analyses of the initial calibration cannot be Performed until the criteria is met.

10.3.2 The GC/MS tuning standard should also be used to assess GC column performance and injection port inertness. DDT breakdown to DDD and DDE should not exceed 20%. Corrective action such as injection port maintenance must be performed when degradation is excessive. The calculation for % breakdown is as follows:

$$\%\ \text{breakdown (DDT)} = \frac{\text{Peak area of (DDE + DDD)} * 100}{\text{Peak area of (DDT+DDE +DDD)}}$$

10.3.3 Measure peak tailing using guidance provided in the USACE Shell document (Appendix 1). Quantitative peak tailing factors of 3.0 and 5.0 are recommended for benzidine and pentachlorophenol respectively. Check also the instrument response for these compounds. Clean the injection port and/or break off the first 10- to 25-cm of the capillary column as needed to restore GC performance.

10.4 Initial Calibration (ICAL) - Once the criteria in sections 10.3.2 and 10.3.33 are met, the analysis of initial calibration standards can be performed. Directly inject the initial calibration in the following order: 2-STD, 5-STD, 10-STD, 20-STD, 50-STD, 70-STD, 100-STD, & 150-STD. The 2-STD and/or 5-STD (optional) are run for projects requiring lower MQLs. The 10-STD is normally used to set MQLs for most compounds with a few exceptions. The calibration curve may be run from high to low instead of low to high as described. Summarize the initial calibration (See Appendix 2 for documentation) data in Form-6 equivalents. After the completion of the 50 STD standard ensure that all compounds have been appropriately identified. A five-point calibration is required for response factor or linear regression method of calibration. Additional points will be required for non-linear regression methods. Use all the points or drop one of the points (either the lowest or the

highest, but not any of the middle points) if curvature is a problem for some of the compounds.

10.5 Tabulate the area response of the characteristic ions against concentration for each compound and each standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard that corresponds to the SVOC compounds is shown in ***TABLE 1***. The RF is calculated as shown below:

$$RF = (A_s C_{is}) / (A_{is} C_s)$$

where:
A_s = Area of the characteristic ion for the compound being measured
A_{is} = Area of the characteristic ion for the specific internal standard
C_{is} = Concentration of the specific internal standard
C_s = Concentration of the compound being measured

10.6 The average RF must be calculated and recorded for each compound using five to seven RF values calculated from the initial calibration analysis.

10.7 A system performance check (SPCC) is used to evaluate the calibration curve. Four compounds are checked for minimum average relative response. These SPCC compounds are N-nitroso-di-propylamine; hexacholorocyclopentadiene; 2, 4-dinitrophenol; and 4-nitrophenol with the minimum relative RF being 0.05. If the minimum RF for these semi-volatile SPCCs are below 0.05, the system must be evaluated and corrective action must be taken. There are a variety of problems such as standard mixture degradation, injection port inlet contamination, contamination at the front end of the column and active sites in the column or chromatographic system. The SPCC compounds for minimum RF must be met before proceeding.

10.8 Using the RFs from the initial calibration, calculate and record the percent relative standard deviation (%RSD) for all the compounds. The percent RSD is calculated as follows:

$$\%RSD = ((SD)/(AVER\ RF))*100\%$$

where:
AVE RF = mean of the 5 (or more) initial RFs for a compound
SD = standard deviation of the 5 (or more) initial RFs for a compound.

The percent relative standard deviation should be less than 15% for all the target compounds. However, before any analytical samples can be analyzed, the % RSD for each individual Calibration Check Compound (CCC) must be less than 30%. The CCCs are listed in ***TABLE 4***.

10.9 If a %RSD greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column active sites is required before reattempting the calibration. The calibration curve must be re-analyzed if any CCC remains above 30% RSD.

10.10 Linearity - If the % RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range and the average relative response factor may be used for quantitation. The analyst, exercising professional judgment, may use regression methods for compounds with less than 15% RSD, if decreasing or increasing trends are observed for response factors at various concentrations. **NOTE:** The maximum allowable %RSD for any compound is 40% according to Appendix I of the USACE document EM 200-1-3.

10.11 If the % RSD of any detected compound is greater then 15%, use least square regression methods to calculate sample concentrations. For regression methods, the instrument response is treated as the dependent variable (y) and the standard amount is the independent variable (x). When "Cs" is used as "x", use "As*Cis/Ais" as "y". When "Cs/Cis" is used as "x", use "As/Ais" as "y". Do not include origin (0,0) as an extra calibration point.

10.12 Weighted least squares method is preferred for calculating concentrations more accurately at lower levels. A linear least squares equation ($y = a*x + b$) is preferred over non-linear calibration equations ($y = a*x^2 + b*x + c$). The coefficient of determination (COD or r^2) or correlation coefficient (r) should be used to measure the goodness of fit. For regression models to be used for quantitative purposes, the COD or r^2 must be greater than or equal to 0.99 and the correlation coefficient (r) should be greater than 0.995. A value of 1.00 for COD and/or "r" indicates a perfect fit.

10.13 Corrective action may be required if the criteria for %RSD, r, r2, or COD are not met.

10.13.1 An initial calibration (ICAL) should be considered a single event process. Consult the QA office or check applicable EPA methods (e.g. SW-846 Method 8000C and 8270D) for additional guidance on appropriate corrective action.

10.13.2 Re-fitting of ICAL – The selected model for ICAL may be subjected to an additional re-fitting check to establish representativeness of the model for each compound in each ICAL standard. This is an optional procedure, which is recommended but not required since automated data processing procedures may not be able to accommodate it. Calculate the %D between the calculated amount (Cc) for each compound in each ICAL standard from the ICAL model and its expected value (Ce). Ideally, the %D ((Cc-Ce) * 100 /Ce) for each compound in ICAL standard should meet the QC criteria for %RSD. It is possible for one or more ICAL standards to have %D exceeding the %RSD criteria, while the %RSD for ICAL model is within QC limits. The re-fitting procedure may be used to identify ICAL standards that need re-analysis or dropped from the calculation of ICAL model when re-analysis was not performed.

10.13.3 Check and optimize instrument operating conditions including automated peak integration procedures for appropriateness in addressing the chromatographic needs of the specific compound with problems.

10.13.4 If the problem appears to be associated with just one of the ICAL standards, that one standard may be reanalyzed once immediately (must be within 8-hours) after the ICAL. Discard all the results from the original analysis of the standard in question and replace it with the results from re-analysis.

10.13.5 When additional calibration standards (six or seven instead of five) are analyzed, narrowing the range of concentrations to be used for determining the goodness of fit is an acceptable procedure. Five or more standards should remain for the calculation of RSD or linear regression for each compound after one or more points are removed from the ICAL. More points (6 for second order and 7 for third order) may be needed for non-linear regression. It is generally prohibited to remove data points from within a calibration range, while still retaining the extreme ends of the calibration range. Do not discard a middle point unless it appears to have an obvious problem and six or more standards were analyzed for ICAL and re-analysis of the affected standard was not performed within the required time limits. Changes to the upper end of the calibration range will affect the need to dilute samples above the range. A change to the lower range is not recommended since it will affect the method quantitation limit (MQL) used to report data. The default MQLs, which are based on the lowest ICAL concentration are stored in the LIMS and project-specific changes are not practical.

10.13.6 The refitting procedures may lead to failed %Ds for upper level calibration standards for average response factor methods. The analyst may take alternate corrective actions such as the analysis of sample extracts under dilution for the affected compounds.

10.13.7 The refitting procedures may lead to failed %Ds for lower level calibration standards for regression methods. The analyst may take appropriate corrective action such as flagging the sample result as an estimated amount ("J" flag) at the lower level encountered in the sample.

10.14 Grand mean RSD – Previous versions of the EPA method(s) had provisions for the calculation of the grand mean %RSD for all target compounds in the ICAL solution, when the % RSD of any detected compound is greater than 15%. Under this option (#2 in DoD QSM, Version 2, Appendix DoD-B, Table B-3), the grand mean %RSD should be <15% with no individual analyte RSD >30% for ICAL to be acceptable for all the compounds. This option for ICAL should be discontinued.

10.15 The term "calibration verification" (CALV) is used to describe initial verification (ICV) of the initial calibration (ICAL) using second-source standards and subsequent continuing verifications (CCVs) using same-source standards. The response factors calculated during CALV are not used for sample quantitation since CALV standards are not used as "continuing calibration" standards permitted in some of the EPA methods.

10.16 Initial Calibration Verification (ICV) - Analysis of a second source ICV standard is required before any samples can be analyzed for USACE projects. Since second source standards are not employed for the analysis of LCS/LCSD and MS/MSD samples, ICV analysis is applicable for all projects. Calculate the percent difference (%D) or %Drift for the observed response factor in ICV from the mean RF as described in Section 10.23. The %D or %drift should not exceed 25% for each target compound. Document calibration verification in instrument printouts as shown in Appendix 2. Check the ICAL and ICV standards if it is not achievable for all the target compounds without any exception. Use professional judgment for non-USACE projects and for compounds that are not contaminants of concern (COC) for USACE projects. The ICV may be used as the beginning CCV for sample analysis.

10.17 If the above criteria are met, the analysis for analytical samples can begin.

10.18 Tuning verification - Prior to the analysis of samples outside the 12-hour time limit of the initial calibration (Table 2), the criteria for the tuning solution must be met. These criteria must be demonstrated every 12-hours.

4.1 Calibration verification (CALV) – The initial calibration curve for each compound of interest must be checked and verified once every 12-hours with a calibration verification standard. Calibration verification is not necessary for samples analyzed immediately after ICAL provided they are within 12-hours of the tune standard analyzed with ICAL. Calibration verification includes initial calibration verification (ICV) and continuing calibration verification (CCV). Use standard SW-846 procedures for ICVs and CCVs.

10.19 Analyze the 50-STD. Summarize CCV in Form-7 equivalents (See also Appendix 2 for documentation).

10.20 Check the SPCC compounds for minimum RF requirements. If the minimum RF for these semi-volatile SPCCs are below 0.05, the system must be evaluated and corrective action must be taken.

10.21 After checking the SPCC compounds, calculate either the percent difference (%D) (for response factors) or percent drift (%D) (for linear regression) for the CCCs using one of the following equations:

$$\%D = \%Difference = ((RF_v - RF_m) * 100)/(RF_m)$$

$$\%D = \%\ drift = ((C_v - C_m) * 100) / (C_m)$$

where:

RF_m = Average or mean response factor from ICAL
RF_v = Observed RF for the CALV standard
C_m = True concentration of the CALV standard.
C_v = Measured concentration of the CALV standard using selected quantitation method (e.g. linear regression).

10.22 If the %D for each CCC is less than 20%, the initial calibration is assumed to be valid. If the criterion is not met (i.e., >20%), for any one of the CCCs, corrective action must be taken. If the source of the problem cannot be detected, then a new five/six point calibration must be generated. This criterion must be met before any quantitative analysis can begin. Check also the %D for all the compounds.

10.23 For specific USACE projects, client may specify that %D for the contaminants of concern (COC) should be within the CALV QC limits (<20%).

10.24 Grand mean %D – The procedure described for the calculation of grand means in previous versions of the EPA SW-846 method(s) for the %D of all target compounds in the CALV solution, when the %D of any detected compound is greater than 20%. According to that discontinued procedure, the grand mean %D should be <20% with no individual compounds except CCCs exceeding 25% for CALV to be acceptable for all the compounds. This procedure should be discontinued.

10.25 The internal standard responses and retention times in the check calibration standard must be evaluated immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration check (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made, as required.

10.26 If the EICP area for any of the internal standards (in all analyses) changes by a factor of two (-50% or + 100%) from the last daily calibration check, the mass spectrometer must be inspected for malfunctions and corrections must be made as necessary. If any samples were analyzed after the system checks are invalid, those samples will need to be re-analyzed after corrections have been made.

11 ANALYSIS OF EXTRACTED SAMPLES

11.1 Once the samples have been extracted and all system performance checks have been established as stated in the previous sections, sample extracts can be analyzed.

11.2 Each 1-mL aliquot is spiked with 10-uLs of internal standard solution (Ultra Scientific, Catalog number US-108N, concentration 4000 ug/mL). The final concentration of internal standard is 40-ug/mL for each 1-mL extract. The sample extracts and associated quality control sample (MB, LCS, LCSD, MS, and MSD) extracts are analyzed in the same manner as calibration standards. Document analysis on instrument logs (See Appendix 13 for documentation).

11.3 All analyses are performed during an analysis sequence. The sequence begins with instrument tune, which is followed by the verification of calibration (CCV). The sequence ends when the set of samples (MB, LCS, LCSD, MS, MSD, and field samples) has been injected within 12-hours following the instrument tune, which is performed every 12 hours.

11.4 The preferred approach is to analyze the batch QC samples not containing matrix (e.g. MB, LCS, and LCSD) immediately after the analysis of CCV. The matrix QC samples (unspiked sample, MS, and MSD) may be analyzed next. The associated field samples may be analyzed immediately thereafter. Analysis of all the samples in a QC batch may not be possible and it is not required.

11.5 If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, reanalyze sample extracts after appropriate dilutions. Additional internal standard must be added to the diluted extract to maintain the required 40 ng/uL level for each internal standard in the extract that is injected. The diluted extract must be reanalyzed.

11.6 If surrogate recovery results exceed the QC limits, reanalyze sample extracts after appropriate cleanup or dilution, as necessary. Additional internal standard must be added to the diluted extract to maintain the required 40 ng/uL level for each internal standard in the extract that is injected. The diluted extract must be reanalyzed.

11.7 If internal standard areas exceed the QC limits, reanalyze sample extracts after appropriate cleanup or dilution, as necessary. Additional internal standard must be added to the diluted extract to maintain the required 40 ng/uL level for each internal standard in the extract that is injected. The diluted extract must be reanalyzed.

12 DATA INTERPRETATION

12. 1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum with characteristic ions in reference mass spectrum. The reference mass spectra are established by generating them from the analyses performed during initial calibration. The characteristic ions are defined to be the three ions of the greatest relative intensity or any ions over 30% relative intensity if less then three such ions occur in the reference spectra. Compounds should be identified as present when the criteria below (See also Appendix 4 for documentation) are met.

12.1.2 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

12.1.3 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

12.1.4 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

12.1.5 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different retention times.

12.1.6 Identification can become difficult when more than one analyte elutes chromatographically causing overlapping of spectra. It is still possible to identify a compound by closely examining the EICP of the appropriate ions. Each analyte spectrum will contain extraneous ions contributed by the co-eluting compounds.

12.2 Tentatively Identified Compounds - A library search may be necessary when additional compounds are present that are not identified as target analytes. Guidelines for making tentative identification (See also Appendix 5 for documentation) are:

12.2.1 Relative intensities of major ions in the reference spectrum should be present in the sample spectrum.

12.2.2 The relative intensities of the major ions should agree within ± 20%.

12.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.4 Ions present in the sample but not in the reference should be reviewed for possible background contamination presence of coeluting compounds.

12.2.5 Ions present in the reference but not in the sample should be reviewed for possible subtraction from sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create discrepancies.

13 QUANTITATIVE ANALYSIS

13.1 The internal standard technique is used to determine the concentration of an identified target compound. For water samples the calculation is as follows:

$$\text{Concentration } (\mu g/L) = \frac{(A_s) * (IS) * (V_e)}{(A_{is})\,(RF_m)\,(V_s)}$$

where:

A_s = Area of characteristic ion for compound being measured.
IS = Concentration of the internal standard in the extract injected (µg/mL)
Ais = Area of characteristic ion for the internal standard
RF_m = Mean Response factor for compound being measured
V_s = Volume of water (L) extracted
V_e = Volume of the final extract (mL), taking into account any dilutions made

For soil samples the calculation is as follows:

$$\text{Concentration } (\mu g/kg) = \frac{(A_s) * (IS) * (V_e) * (100)}{(A_{is}) * (RF_m) * (W_s) * (\%S)}$$

where:

A_s = Area of characteristic ion for compound being measured.
IS = Concentration of the internal standard in the extract injected (µg/mL)
Ais = Area of characteristic ion for the internal standard
RF_m = Mean Response factor for compound being measured
W_s = Wet weight of soil sample (kg) extracted
V_e = Volume of the final extract (mL), taking into account any dilutions made
%S = % Solids.

13.2 See Appendix 3 for documentation of quantitation reports and chromatograms.
13.3 Tentatively Identified Compounds (TICs) - Sometimes it is necessary to give an estimated quantitation value for a non-target analytes. The equations described above can be used to calculate sample amounts, substituting 1.0 for the RF_m and using the total ion areas in the chromatograms for the unknown and the nearest IS for the unknown. The result should be identified as an "estimated value only" by following the value with a "J" code. The report should also identify which IS was used to determine the concentration.

14 QUALITY CONTROL

This section will provide guidance when evaluating and validating data generated by this SOP. It provides procedures to assess if data produced is valid and legally defensible. Include a checklist cover page for the raw data package and document review in a draft case narrative, while performing the review in accordance with the review checklist (See Appendix 7). Verify that sample preparation logs are complete and the instrument run logs are available for review. Anomalies and exceptions should be documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on these checklists. USACE projects may have special requirements that will require extra attention. A simple, universal procedure for evaluating choosing appropriate corrective action doe not exist to deal with situations when one or more QC parameters are outside the acceptance window. The objective of

the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints.

14.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) projects may have additional requirements that are related to USACE laboratory validation (CX), Department of Defense Quality Systems Manual (DoD QSM), USACE District requirements, government contract requirements, specific USACE project requirements or USACE Architect/Engineer (A/E) requirements. USACE-CX requirements (Summary of Method Quality Objectives for Method 8260B), DoD QSM requirements, and AFCEE requirements are included in Appendix 8.

14.2 The USACE requirements shown in Appendix 8 and elsewhere in this SOP are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may be unattainable for routine sample analysis. Since USACE MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. The laboratory may employ default QC limits based on a combination of DoD QSM limits, USACE Shell limits, laboratory statistical limits, or other administrative limits. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

14.3 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality objectives and/or other QC requirements. Exceptions and data qualifications necessary should be documented on the checklists and eventually on case narratives.

14.4 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist (Appendix 8), perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete. For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete. For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director will review the checklists (initial/date as necessary), prepare case narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

14.5 The blank criteria shown in Appendix 8 has been modified by USACE auditor(s) as one half of the applicable MQL (personal communications, USACE auditor, 1999).

14.6 Summarize sample results using sample result Form 1 equivalents (See Appendix 12). Use the same format for all analyses including QC samples such as MB, LCS, LCSD, MS, and MSD. Evaluate acceptability of analysis results using Form-8 internal standard

area and retention time summary (See Appendix 9). The MDLs, LLRs, or RLs (when applicable), and MQLs applicable to each matrix and/or method are shown in Tables 6 and 7 and also in Appendix 6. Report positive results greater than the MDL and/or the LLR or RL but less than the MQL as estimated with a "J" flag. Report positive results that are greater than the upper calibration limit as estimated with an "E" flag. Evaluate sample data as shown in the Data Verification section and apply additional qualifiers as needed. Document them in the checklists (Appendix 7) and in the case narrative provided to clients. Report sample results to the client in reports shown in Form-1 equivalents (Appendix 12).

14.7 Summarize QC sample results for MS/MSD in Form-1 in the same manner as sample results and the recoveries in Form-3 equivalents (Appendix 11). Evaluate them as shown in the Data Verification section.

14.8 Summarize QC results for LCS/LCSD in Form-1 in the same manner as sample results and the recoveries in Form-3 equivalents (Appendix 10). Evaluate them as shown in the Data Verification section.

14.9 Summarize surrogate recoveries in Form-2 equivalents (Appendix 12). Evaluate them as shown in the Data Verification section.

14.10 Summarize method blank (MB) results in the same manner as sample results (Appendix 12).

14.11 Summarize MB sample results in Form-1 in the same manner as field sample results. Document MB or QC association in Form-4 equivalents (Appendix 12). Evaluate MB as shown in the Data Verification section.

15 **DATA VERIFICATION AND VALIDATION -** This section will provide guidance when evaluating and validating data generated by this SOP.

15.1 HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

15.1.1 WATER - The holding times for semi-volatiles is 7 days from sampling.

15.1.2 SOIL – A 14 day holding is used for soil and waste samples.

15.1.3 TCLP leaching should be performed within 14 days of waste sample collection. TCLP leachates (aqueous samples) must be extracted within 7 days of leachate preparation.

15.1.4 All sample extracts should be analyzed within 40 days after extraction.

15.1.5 Holding times are established by comparing the chain of custody sampling date with the date of the injection after purging. TCLP holding times must be established by comparing first the COC to the leaching date and then the leaching date to the injection date.

15.1.6 ACTION – If holding times have been exceeded then all positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or invalid (I).

15.2 INSTRUMENT TUNING/PERFORMANCE – Tuning and performance criteria for mass resolution, identification and sensitivity should meet criteria established in ***TABLE 2***. DDT breakdown to DDD and DDE should not exceed 20%. Peak tailing factors for benzidine and pentachlorophenol should be below 3.0 and 5.0 respectively. Perform appropriate corrective action as described in this SOP to restore chromatographic performance to acceptable levels.

15.2.1 Compare mass calibration from tabular report in the raw data with each criteria form.

15.2.2 Check to see if the tuning report is present for each 12-hour period of samples analyzed.

15.2.3 Verify that the spectrum is present.

15.2.4 Calculate % abundance.

15.2.5 ACTIONS – If mass calibration is outside criteria, recalibrate and rerun tuning standard for an acceptable tune report. Reanalyze all associated samples from failed tune. If mass calibration is outside criteria, all associated data may be qualified as estimated (J) or invalid (I) by the client at client's discretion.

15.3 INITIAL CALIBRATION (ICAL) – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the mass spectrometer is capable of producing data within a range that is linear. See also additional client-specific initial calibration requirements including second-source ICV analysis (Appendix 8).

15.3.1 SPCC – Check to see that the (SPCC) compounds are within acceptable criteria – (RRF > 0.005).

15.3.2 ACTION - If any SPCC compound does not meet the minimum criteria as stated, then correct the underlying problem and re-analyze the ICAL. The calibration is considered invalid.

15.3.3 CCC – Check to see that the CCC compounds are within acceptable criteria (% RPD < 15 %) for each individual Calibration Check Compound.

15.3.4 ACTION – If any CCC compound does not meet the minimum criteria as stated, then correct the underlying problem and re-analyze the ICAL. The calibration is considered invalid.

15.3.5 Other compounds – Check to see if any other compounds are outside the 15% criteria or less for %RSD. If the %D for a compound is outside the 15% criteria, check to see if regression methods are used. The goodness of fit should be measured with the coefficient of determination (COD) or related quantities (r^2 and r) greater than 0.99.

15.3.6 ACTION – Positive results for compounds not meeting the criteria may need to be qualified as estimated (J) by the user (client). Report detects and non-detects without qualifiers and mention these outliners in the checklist and in the case narrative.

15.4 INITIAL CALIBRATION VERIFICATION – Calibration verification includes initial calibration verification (ICV) and continuing calibration verification (CCV). A second

source ICV is used to check the integrity of the ICAL standards used for calibrating the instrument. The 12-hour analytical shift begins with the injection of the tuning standard (DFTPP) if a separate standard is used instead of including the DFTPP in ICV. For samples analyzed immediately following the ICAL, the ICV is used to check for internal standard areas and retention times.

15.4.1 13.5.1 Target Compounds - Check to see if %D for any of the target compounds is outside the USACE QC criteria (<30%).

15.4.2 13.5.2 ACTION - Check the ICAL and ICV standards for integrity. Replace one or more of the working standards (ICV first and ICAL later) for trouble shooting. Correct the problem(s) and repeat ICV analysis. Use professional judgment for non-USACE projects.

15.4.3 13.5.3 SPCC – Check to see that the (SPCC) compounds are within acceptable criteria as described for CCVs.

15.4.4 13.5.4 CCC – Check to see that the CCC compounds are within acceptable criteria as described for CCVs.

15.4.5 13.5.5 ACTION - If any SPCC compound does not meet the minimum criteria as stated in the calibration section, then correct the underlying problem and re-analyze the ICV. If the ICV passes, the ICAL is considered valid. Otherwise, the initial calibration (ICAL) is considered invalid. A new ICAL should be run.

15.4.6 13.5.6 ACTION – If any CCC compound does not meet the minimum criteria as stated in the calibration section, then correct the underlying problem and re-analyze the ICV. If the ICV passes, the ICAL is considered valid. Otherwise, the initial calibration (ICAL) is considered invalid. A new ICAL should be run.

15.4.7 13.5.7 ACTION - When ICAL and ICV are acceptable based on SW-846 criteria, positive results and non-detects for target compounds not meeting the 30% %D may be reported without data qualifications for non-USACE projects. The ICAL and ICV standards should be inspected and replaced, if necessary for compliance with this requirement for USACE projects.

15.5 CONTINUING CALIBRATION – The continuing calibration is used to check that the instrument is still operating under the linear range of the initial calibration and has remained stable every 12 hours. See also additional client-specific calibration verification requirements (Appendix 8).

15.5.1 SPCC – Check to see that the (SPCC) compounds are within acceptable criteria – (average RRF > 0.005).

15.5.2 ACTION - If any SPCC compound does not meet the minimum criteria as stated, then correct the underlying problem and re-analyze the CCV. The ICAL is considered invalid unless the SPCC criteria are met. A new ICAL may need to be run.

15.5.3 CCC – Check to see that the CCC compounds are within acceptable criteria – (% D < 20%).

15.5.4 ACTION – If any CCC compound does not meet the minimum criteria as stated, then correct the underlying problem and re-analyze the CCV. The

ICAL is considered invalid if the CCC criteria are not met. A new ICAL may need to be run, if necessary.

15.5.5 Other compounds – Check to see if any other compounds have %D outside the 20% criteria.

15.5.6 ACTION – Positive results for samples not meeting the criteria for the CCC and SPCC criteria may be qualified as estimated (J) or invalid (I) by the user (client) at their discretion. Any positive results for compounds other than CCC and SPCC not meeting the criteria may be qualified as estimated (J) by the user (client). Report data without qualifications documenting the QC outliers in the checklist and in the case narrative.

15.6 METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. The processing of blanks helps eliminate reporting of false positives.

15.6.1 Method blanks should be free of all target analytes and other interfering non-target analytes. To be acceptable, blank results should not exceed one half of the PQL for any compounds with the exception of common plasticizers such as phthalate esters.

15.6.2 Review the results of all associated blanks and verify that the method blank has been reported per matrix for each system used to analyze the associated samples. If levels greater than one half the MQLs are present, inspect and correct the problem. Reanalyze all associated samples. The only exception may be quick turn around projects.

15.6.3 ACTION – All positive results are reported with a "B" code if contaminants are found in the method blank.

15.6.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, reanalysis of the samples is not required.

15.6.5 ACTION – Any compound detected in the sample which were also detected in associated method blank both being greater than one half of the MQLs (except phthalate esters) should be re-extracted.

15.6.6 ACTION – If contaminants resulting in GC/MS peak saturation are present, all affected compounds are qualified as invalid (I).

15.7 SURROGATE RECOVERIES – Sample quality control is measured by means of spiking individual samples with system monitoring compounds which are different structurally, but are chemically similar to classes of target compounds. Three base neutral (BN) and three acid (A) surrogates are spiked into the samples just prior to when the extracting solvent is added to the sample. At least two of the BN surrogates and two of the A surrogates should be within the QC limits for sample analysis to be acceptable.

15.7.1 Verify surrogate recoveries by comparing recovery limits shown in ***TABLE 5*** with the observed recoveries in the samples.

15.7.2 ACTION – When one or more of the three BN surrogate and/or one or more of the three acid surrogate recoveries are outside QC limits the sample must

be re-analyzed. Reanalysis may be performed under dilution if original analysis should indicate high levels of target and/or non-target analytes. Otherwise, the sample should be re-extracted and re-analyzed.

15.7.3 ACTION – When the surrogate recoveries are still outside the QC limits after re-analysis or re-extraction/re-analysis, data from both sets of analyses may be reported with recommendations for data usability in the case narrative.

15.7.4 ACTION – When any of the six surrogate recoveries are outside the QC limits for controlled matrices such as MB, LCS and LCSD, it may indicate problems with method performance. Problems should be identified and corrected as soon as possible, preferably prior to the analysis of additional samples. QC criteria described above also applies to these samples.

15.8 LABORATORY CONTROL STANDARD (LCS) – The analysis of LCS samples is used to evaluate method competence in the absence of matrix effects. The LCS is evaluated by comparing the percent recovery for all of the target analytes to the laboratory QC limits, which are identical to the 3-sigma DoD QSM QC limits. See Appendix 10 for guidance on documenting LCS and LCSD concentrations and %R in QC summary report forms. The DoD QSM specifies the number of compounds that can exceed the 3-sigma QC limits based on statistical considerations. The LCS recovery for such compounds should not exceed the marginal exceedance 4-sigma (ME) limits. Evaluation procedures may include comparison of LCS recoveries with those determined by project-specific DQOs (See project QAPP, if available), laboratory statistical control limits, or USEPA guidance. Initially, the effect of QC failure on the samples should be evaluated. Regardless of this assessment, steps shall be taken to find the source of the problem and correct it.

15.8.1 CHECK - Compare the %R results for each compound in LCS with laboratory QC limits to see if all the results are within the acceptable range. Flag outliers with “ME” if it is within the 3-sigma limits. Flag it with “ME*” if it is outside the 4-sigma ME limits.

15.8.2 CHECK - Compare observed %R results for each compound with the laboratory limits for LCS/LCSD recoveries (e.g. USACE criteria). Perform manual calculation for %R, if necessary for QC outliers. Document the number of compounds that exceeded the QC limit and compare it against the maximum recommended by DoD QSM. Document this review in checklists (Appendix 7) and in case narratives.

15.8.3 ACTION - Typically, the LCS would be reanalyzed for the failed analytes (ME*) only. If the second analysis fails, then the LCS, method blank, and all associated samples of the batch would be reanalyzed for the failed analytes only. If sufficient sample is not available for reanalysis, or if the corrective action is ineffective, discuss the situation with the client project manager (or chemist). Include a discussion of the situation in the case narrative. The case narrative should discuss the corrective action taken and any other information.

15.8.4 ACTION - For non-USACE projects, no action may need to be taken on LCS and/or LCSD recovery data alone either to reanalyze samples and/or to qualify associated samples. Providing data of known quality is sufficient for most projects. If significant deviations (e.g. number of target compound

exceeding QC limits for recoveries is greater than the allowable maximum) are noticed, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements, which may be compound specific. Check with the project manager about such requirements. Professional judgment may be used by the user (client) in evaluating sample data qualification options in conjunction with other QC data for the project.

15.9 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike recovery data are used to evaluate the accuracy of the analytical method on project samples. The evaluation is performed by comparing the %R for target analytes to the DoD QSM limits established for the LCS samples. See Tables 7 to 10 and Appendix 11 for guidance on documenting MS and MSD concentrations and percent recoveries in QC summary report forms.

15.9.1 MS data evaluation is more complex than method blank or LCS data evaluation since they are complicated by matrix effects in addition to sample preparation and analysis errors. The heterogeneity of grab soil samples, and potential non-representativeness of sequentially collected water samples, etc. further complicate the evaluation since it is assumed that the native concentrations are constant in triplicate analyses. In addition, concentrations of the target analytes in the sample can also far exceed spike amounts. If the native concentration of target analytes in the sample chosen for spiking is high relative to the spiking concentration, the differences in the native concentration between the unspiked sample and the spiked samples may also become very significant.

15.9.2 In addition to their reluctance to pay for MS and/or MSD analyses, most clients may not know what samples to collect for MS analysis. Clients may not collect sufficient samples, have no knowledge of the native analyte concentrations in the chosen sample, and may not have knowledge of MS spiking levels that are appropriate either for the sample or their project. For procedures with multiple target analytes, spiking levels may be inappropriate for one or more analytes, if samples that contain target analytes are chosen either in the field or by the laboratory. If no project samples are collected for MS analysis, samples from another project used for MS analysis may not be representative of the project samples. When project samples (e.g. surface soil) are chosen, their MS recovery characteristics may be different from those of other samples (e.g. sub-surface soil) in the same batch.

15.9.3 Document MS/MSD recoveries in instrument printouts and in LIMS generated reports as shown in Appendix 11. Flag QC outliers with “ME” and “ME*” on client reports.

15.9.4 CHECK - Compare %R results for each compound with the laboratory limits for MS/MSD recoveries as described for LCS. Perform manual calculation for %R using raw data and extraction sheet information to check outliers. Document this review in checklists (Appendix 7) and in case narratives.

15.9.5 ACTION – The results of the MS are evaluated, in conjunction with other QC information, to determine the effect of the matrix on the bias of the analysis.

For DoD/USACE projects, acceptance criteria may allow for marginal exceedances as described for LCS (See above). Although these criteria are not applicable to LCS data, steps should be taken to find the cause of failure and corrective action to remedy it. Matrix spike recoveries that fail to meet the appropriate acceptance criteria may indicate that a potential matrix effect is present. A review of the MSD result, if available, may confirm the matrix effect, if it is in the same direction and same order of magnitude. Check the LCS and/or LCSD recoveries for affected compounds to rule out laboratory problems.

15.9.6 ACTION - If the sample chosen for MS/MSD is inappropriate, notify the client. When additional payments are authorized for re-analysis, re-extract and/or reanalyze the MS/MSD samples after employing cleanup procedures (if applicable), dilution techniques to minimize matrix interference, or using other project samples that appear to be clean, when sufficient quantities are available. Re-spike the sample at a higher level (e.g., at approximately two to four times the sample concentration), then reanalyze the sample based on project-specific requirements. It is the responsibility of the client at all times to select appropriate samples for MS analysis, make payments for additional analyses, and to offer necessary guidance as needed to meet their data quality objectives.

15.9.7 ACTION - If the matrix effect cannot be resolved, discuss the situation in the case narrative. No action may be taken by the laboratory to qualify associated sample data based on MS/MSD recovery data alone. Professional judgment may be used by the user (client) in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project. Document QC outliers on the checklist, client reports (*) and draw attention to the QC outliers on the case narrative.

15.10 INTERNAL STANDARD PERFORMANCE – Internal Standard (IS) performance criteria (See also Appendix 9) ensures that the GC/MS sensitivity and response is stable during each run.

15.10.1 IS area counts must not vary by more than a factor of 2 (-50% or + 100%) from the associated ICAL/Calibration Verification standard. The internal standard areas for the CCV should not differ by a factor of 2 (-50% or + 100%) from the initial calibration of the same standard.

15.10.2 The retention time of the internal standard must not vary by more than 30 seconds from the associated daily calibration standard.

15.10.3 ACTION – Document IS areas and RT on Form-8 equivalents (Appendix 9). If an internal standard is outside of QC limits of the associated daily standard, then re-analysis is required. Reanalysis under dilution may be required if it appears that interference's occur from target and/or non-target analytes at high concentrations relative to the ICAL. Do not dilute the sample to the point where all target analytes are non-detect. Professional judgement must be used when dilutions are required for IS criteria.

15.10.4 ACTION – If an internal standard is outside of QC limits of the associated ICAL or CCV standard, re-analyze the affected sample(s) first. If re-analysis has been performed and it is still out, it is of the professional judgement of the analyst to dilute or not to dilute the sample further so as to not to dilute out any target analytes. Report the results for the most acceptable (or report both analyses depending on project requirements) analysis and document the problem on the checklist and on the case narrative. The user (client) may qualify positive results for compounds quantitated using the affected IS as estimated (J). If a very low IS area is reported, indicating a loss of sensitivity, non-detects may be qualified as invalid (I) by the user. Document problems and recommendations in the case narrative.

15.10.5 ACTION – If an IS retention time varies by more than +/- 30 seconds, the total ion profile must be checked for any false positives or negatives. If a large shift has occurred, then professional judgement must be used as to whether reanalyze or not.

15.11 TARGET COMPOUND IDENTIFICATION – Criteria for GC/MS qualitative analysis (Section 12.1) have been provided to minimize the number of erroneous positive or negative identifications of compounds.

15.11.1 For positive identification of the target compound, its RT must be within +/- 0.06 relative time (RRT) units of the standard RRT.

15.11.2 Check to see if the mass spectra of the sample compound (See Appendix 4) and a laboratory generated standard reference spectrum match according to the following criteria listed in this SOP.

15.11.3 For tentatively identified compounds identification and quantitation, see Section 12.2 (See also Appendix 5) for guidelines.

15.11.4 ACTION – Professional judgement must be used when applying the criteria for GC/MS analysis of target analytes. The ion pattern and clusters of the extracted ion profile should match fairly well, taking into account factors such as matrix ions and column bleed.

16.0 REFERENCES

16.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 4, January 1998.

16.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), December 1996.

16.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.

16.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

16.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3500B, Organic Extraction and Sample Preparation, December 1996.

16.6 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3520C, Continuous Liquid-Liquid Extraction, December 1996.

16.7 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3510C, Separatory Funnel Liquid-Liquid Extraction, December 1996.

16.8 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3511, Organic compounds in water by microextraction, Revision 0, November 2002.

16.9 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3540C, Soxhlet Extraction, December 1996.

16.10 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3541, Automated Soxhlet Extraction, December 1996.

16.11 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3545, Pressurized Fluid Extraction, December 1996.

16.12 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3550B, Ultrasonic Extraction, December 1996.

16.13 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3550C, Ultrasonic Extraction, Draft Revision 3, March 1999.

16.14 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3570, Microscale Solvent Extraction (MSE), Revision 0, November 2002.

16.15 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3600C, Cleanup, December 1996.

16.16 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3640A, Gel-Permeation Cleanup, December 1996.

16.17 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3580A, Waste Dilution, December 1996.

16.18 Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, EPA, 40 CFR Part 136, Method 625: Base Neutral and Acid Compounds, October 1984.

16.19 Methods for the Determination of Organic Compounds in Drinking Water, Supplement II, EPA/600/4-88-039, Method 525.1, Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry, July 1991.

16.20 USEPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-media, Multi-concentration, OLM02.0.
16.21 USEPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-media, Multi-concentration, OLM03.0.
16.22 USEPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-media, Multi-concentration, OLM04.0.
16.23 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.
16.24 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.
16.25 AML Standard Operating Procedures, Q-Water-RC.
16.26 AML Standard Operating Procedures, Q-WasteMgmt-RD.
16.27 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
16.28 AML Standard Operating Procedures, Q-Controlcharts-RC.
16.29 AML Standard Operating Procedures, Q-Sonicator Tuning-RD.

TABLE 1
CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS FOR SEMI-VOLATILE ORGANIC COMPOUNDS ON DB5 CAPILLARY COLUMN

ANALYTE	RETENTION TIME (minutes)	INT STD	MQL (Water) (µg/L)	MQL (soil) (µg/kg)
bis (2-chloroethyl) ether	4.05	1	10	333
Phenol	4.00	1	10	333
2-chlorophenol	4.07	1	10	333
1,3-dichlorobenzene	4.32	1	10	333
1,4-dichlorobenzene	4.43	1	10	333
1,2-dichlorobenzene	4.79	1	10	333
bis (2-chloroisporopyl) ether	5.20	1	10	333
2-methylphenol	5.22	1	10	333
hexachloroethane	5.47	1	10	333
N-nitroso-di-n-propylamine	5.51	1	10	333
4-methylphenol	5.60	1	10	333
Nitrobenzene	5.69	2	10	333
Isophorone	6.35	2	10	333
2-nitrophenol	6.50	2	10	333
2,4-dimethylphenol	6.91	2	10	333
bis (2-chloroethoxy) methane	7.11	2	10	333
2,4-dichlorophenol	7.18	2	10	333
1,2,4-trichlorobenzene	7.34	2	10	333
naphthalene	7.43	2	10	333
4-chloroaniline	7.79	2	10	333
Hexachlorobutadiene	8.11	2	10	333
4-chloro-3-methylphenol	9.41	2	10	333
2-methylnaphthalene	9.39	2	10	333
hexachlorocyclopentadiene	10.13	3	10	333
2,4,6-trichlorophenol	10.50	3	10	333
2,4,5-trichlorophenol	10.50	3	10	333
2-chloronaphthalene	10.74	3	10	333
2-nitroaniline	11.29	3	10	333
acenaphthylene	11.94	3	10	333
2,6-dinitrotoluene	14.04	3	10	333
dimethylphthalate	12.44	3	10	333
Acenaphthene	12.53	3	10	333
3-nitroaniline	0.00	3	10	333
2,4-dinitrophenol	12.84	3	10	333
dibenzofuran	13.02	3	10	333
2,4-dinitrotoluene	13.34	3	10	333
4-nitrophenol	13.41	3	10	333
fluorene	14.04	3	10	333
4-chlorophenyl-phenyl ether	14.26	3	10	333
diethylphthalate	14.33	3	10	333
4-nitroaniline	14.38	4	10	333
4,6-dinitro-2-methylphenol	14.51	4	10	333
n-Nitrosodiphenylamine	14.67	4	10	333
4-bromophenyl-phenyl ether	15.68	4	10	333
hexachlorobenzene	15.93	4	10	333

TABLE 1 (Contd.)
CHROMATOGRAPHIC RETENTION TIMES AND METHOD DETECTION LIMITS FOR SEMI-VOLATILE ORGANIC COMPOUNDS ON DB5 CAPILLARY COLUMN

ANALYTE	RETENTION TIME (minutes)	INT STD	MQL (water) (µg/L)	PQL (soil) (µg/kg)
pentachlorophenol	16.56	4	10	333
phenanthrene	16.97	4	10	333
Anthracene	16.97	4	10	333
di-n-butyl phthalate	19.36	4	10	333
Carbazole	17.62	4	10	333
Fluoranthene	20.42	4	10	333
Pyrene	21.03	5	10	333
butylbenzylphthalate	23.80	5	10	333
benzo (a) anthracene	24.89	5	10	333
Chrysene	24.89	5	10	333
3,3-dichlorobenzidine	25.00	5	10	333
bis (2-ethylhexyl) phthalate	25.88	5	10	333
Di-n-octyl phthalate	27.66	6	10	333
Benzo (b) fluoranthene	27.91	6	10	333
Benzo (k) fluoranthene	27.91	6	10	333
Benzo (a) pyrene	28.63	6	10	333
Indeno (1,2,3-cd) pyrene	31.88	6	10	333
Dibenzo (a,h) anthracene	31.47	6	10	333
Benzo (g,h,i) perylene	31.88	6	10	333
INTERNAL STANDARDS				
1. 1,4-Dichlorobenzene-d4	4.38			
2. Naphthalene-d8	7.38			
3. Acenaphthalene-d10	12.44			
4. Phenanthrene-d10	16.79			
5. Chrysene-d12	24.82			
6. Perylene-d12	28.81			
SURROGATES				
2-Flouorophenol	2.75	1		
Phenol-d5	5.00	2		
Nitrobenzene-d5	5.47	3		
2-fluorobiphenyl	10.25	4		
2,4,6-tribromophenol	14.80	5		
Terphenyl-d14	22.0	6		

NOTE: The retention times and MQLs are dynamic parameters that are subject to change.

TABLE 2
DFTPP MASS-INTENSITIES SPECIFICATIONS

MASS	INTENSITY REQUIRED (RELATIVE ABUNDANCE)
51	30-60% of mass 198
68	<2% or mass 69
70	<2% or mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9 5 mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

TABLE 3
PREPARATION OF WORKING STANDARDS FROM STOCK STANDARDS

Working Stds prep for BNA Stds.

Analytes	Ultra Scientific Catalog No.	Lot No.	Conc. ug/mL	Code
Internal Standard	US-108N	M-1809	4000	A
BNA std	SVM-8270	P-0689	1000	B
Surrogate	ISM-335N	L-629A	1500/1000 ug/ml	C
Diphenylamine	EPA-1118	E-0529A	5000	D

Procedure for preparing calibration curve:

Concentration (ug/ml)	Stock BNA STD 1000 ug/ml (uls)	Stock Int. Std. 4000 ug/ml (uls)	Stock Surrogate (1500/1000ug/ml) (uls)	MeCl2 (uls)	Diphenylamine 5000 ug/ml (uls)
10	10	10	10	968	2
20	20	10	30	936	4
50	50	10	50	880	10
70	70	10	60	846	14
100	100	10	80	790	20
150	150	10	100	710	30

Acid Surrogate Conc. (ug/ml)	BN Surrogate Conc. (ug/ml0
15	10
45	30
75	50
90	60
120	80
150	100

Procedure for MS, & MSD spiking solution (and LCS/LCSD spiking solution using second source) is as follows: 1-ml of BNA (stock conc.-1000 ug/ml) diluted to 20-ml with MeCl2.
Final spiking solution concentration: 50-ug/ml for a spike volume of 1-ml per liter of aqueous sample.

NOTE: Procedures described above are dynamic processes that are subject to change.

TABLE 4
CALIBRATION CHECK COMPOUNDS

BASE/NEUTRAL FRACTION	ACID FRACTION
Acenaphthene	4-chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-dichlorophenol
Hexachlorobutadiene	2-nitrophenol
n-nitrosodiphenylamine	Phenol
di-n-octyl phthalate	Pentachlorophenol
Fluoroanthene	2,4,6-trichlorophenol
benzo(a)pyrene	
Diphenylamine	

TABLE 5
SURROGATE SPIKE RECOVERY LIMITS
WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compound	Control Limits for Percent Recovery (from DoD QSM)			
Matrix:	Water		Soil / Sediment	
	Lower	Upper	Lower	Upper
2-Flourophenol	20	110	35	105
phenol-d5	10	115	40	100
nitrobenzene-d5	40	110	35	100
2-fluorobiphenyl	50	110	45	105
2,4,6-tribromophenol	40	125	35	125
terphenyl-d14	50	135	30	125
	ME Limits		ME Limits	
Phenol-d5	0	135	NA	NA

NOTE: ME = Marginal Exceedance Limits

TABLE 6
Aqueous MDLs, MQLs, and QC Limits for LCS and MS

Semivolatile Organic Compounds (SVOCs) Analysis by GC/MS														
Analytical Management Laboratories														
Matrix: Water	Preparation Method			3510C										
	Analytical Method			8270D										
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**			**DoD QSM Control Limits LCS & MS**		**DoD QSM ME Limits LCS & MS**		**RPD**
Compound	Amount	µg/L	µg/L	**µg/L**	RD =B/A	flag	**µg/L**	RQ =C/A	Flag	Lower	Upper	Lower	Upper	Limit	
	µg/L	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%	
Bis(2-Chloroethyl)ether	5	4.03	0.73	**2.20**	1.8		10	4.5		35	110	25	120	50	
Bis(2-chloroisopropyl)ether	5	3.77	0.66	**1.98**	1.9		10	5.1		25	130	10	150	50	
2-Chlorophenol	5	4.15	0.81	**2.44**	1.7		10	4.1		35	105	25	115	50	
1,3-Dichlorobenzene	5	3.60	0.92	**2.75**	1.3		10	3.6		30	100	20	110	50	
1,4-Dichlorobenzene	5	3.56	0.88	**2.64**	1.3		10	3.8		30	100	20	110	50	
1,2-Dichlorobenzene	5	3.60	0.82	**2.46**	1.5		10	4.1		35	100	20	115	50	
Hexachloroethane	5	3.14	0.83	**2.49**	1.3		10	4.0		30	95	15	105	50	
2-Methylphenol (o-Cresol)	5	3.90	0.63	**1.88**	2.1		10	5.3		40	110	25	120	50	
3/4-Methylphenol (m/p-Cresol)	5	3.79	0.52	**1.55**	2.4		10	6.4		30	110	20	125	50	
N-Nitroso-di-n-propylamine	5	3.97	0.59	**1.78**	2.2		10	5.6		35	130	20	145	50	
Phenol	5	3.48	0.46	**1.39**	2.5		10	7.2		0	115	0	135	50	
Bis(2-chloroethoxy)methane	5	3.85	0.54	**1.62**	2.4		10	6.2		45	105	35	115	50	
4-Chloroaniline	5	0.88	0.29	**0.98**	0.9	*	10	10.2		15	110	10	125	50	
4-Chloro-3-methylphenol	5	3.91	0.40	**1.20**	3.2		10	8.3		45	110	35	120	50	
2,4-Dichlorophenol	5	3.83	0.52	**1.56**	2.5		10	6.4		50	105	40	115	50	
2,4-Dimethylphenol	5	3.25	0.54	**1.62**	2.0		10	6.2		30	110	15	125	50	
Hexachlorobutadiene	5	3.82	0.87	**2.62**	1.5		10	3.8		25	105	15	115	50	
Isophorone	5	4.86	0.61	**1.83**	2.7		10	5.5		50	110	40	125	50	
2-Methylnaphthalene	5	4.13	0.61	**1.82**	2.3		10	5.5		45	105	35	115	50	
Naphthalene	5	3.98	0.73	**2.19**	1.8		10	4.6		40	100	30	115	50	
Nitrobenzene	5	4.08	0.75	**2.26**	1.8		10	4.4		45	110	35	120	50	
2-Nitrophenol	5	5.07	0.39	**1.17**	4.3		10	8.6		40	115	25	125	50	
1,2,4-Trichlorobenzene	5	3.72	0.71	**2.13**	1.7		10	4.7		35	105	25	120	50	
Acenaphthene	5	4.18	0.49	**1.47**	2.8		10	6.8		45	110	35	120	50	
Acenaphthylene	5	4.23	0.50	**1.51**	2.8		10	6.6		50	105	40	115	50	
2-Chloronaphthalene	5	4.13	0.54	**1.62**	2.5		10	6.2		50	105	40	115	50	
4-Chlorophenyl-phenylether	5	4.34	0.47	**1.40**	3.1		10	7.1		50	110	40	120	50	
Dibenzofuran	5	4.31	0.42	**1.26**	3.4		10	7.9		55	105	45	115	50	
Diethylphthalate	5	4.53	0.37	**1.12**	4.0		10	8.9		40	120	30	130	50	
Dimethylphthalate	5	4.42	0.43	**1.28**	3.5		10	7.8		25	125	10	145	50	
2,4-Dinitrophenol	10	6.8	0.51	**1.59**	4.3		10	6.3		15	140	10	160	50	
2,6-Dinitrotoluene	5	6.86	0.42	**1.26**	5.4	*	10	7.9		50	115	35	130	50	
2,4-Dinitrotoluene	5	6.41	0.39	**1.17**	5.5	*	10	8.6		50	120	40	130	50	
Fluorene	5	4.05	0.43	**1.29**	3.1		10	7.7		50	110	40	120	50	
Hexachlorocyclopentadiene	5	5.14	0.35	**1.10**	4.7		10	9.1		25	105	15	115	50	
2-Nitroaniline	5	5.71	0.43	**1.30**	4.4		10	7.7		50	115	35	125	50	
3-Nitroaniline	5	2.70	0.17	**0.56**	4.8		10	17.9		20	125	10	145	50	

TABLE 6 (contd.)
Aqueous MDLs, MQLs, and QC Limits for LCS and MS

Semivolatile Organic Compounds (SVOCs) Analysis by GC/MS															
Analytical Management Laboratories															
Matrix: Water	Preparation Method			3510C											
	Analytical Method			8270D											
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**			**DoD QSM Control Limits LCS & MS**		**DoD QSM ME Limits LCS & MS**		**RPD**
Compound	Amount	µg/L	µg/L	**µg/L**	RD =B/A	flag	**µg/L**	RQ =C/A	Flag	Lower	Upper	Lower	Upper	Limit	
	µg/L	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%	
4-Nitroaniline	5	6.48	0.05	**0.16**	40.2	*	10	62.1		35	120	20	130	50	
4-Nitrophenol	5	6.67	0.37	**1.12**	6.0	*	10	8.9		0	125	0	145	50	
2,4,6-Trichlorophenol	5	3.86	0.42	**1.26**	3.1		10	8.0		50	115	40	125	50	
2,4,5-Trichlorophenol	5	3.78	0.39	**1.18**	3.2		10	8.5		50	110	40	120	50	
Anthracene	5	4.36	0.53	**1.58**	2.8		10	6.3		55	110	45	120	50	
Phenanthrene	5	4.61	0.46	**1.37**	3.4		10	7.3		50	115	40	130	50	
4-Bromophenyl-phenylether	5	4.57	0.49	**1.46**	3.1		10	6.9		50	115	40	125	50	
2-Methyl-4,6-dinitrophenol	25	12.2	0.74	**2.33**	5.2	*	10	4.3		40	130	25	145	50	
Di-n-butylphthalate	5	5.34	0.44	**1.31**	4.1		10	7.6		55	115	45	125	50	
Fluoranthene	5	4.50	0.42	**1.26**	3.6		10	7.9		55	115	45	125	50	
Hexachlorobenzene	5	4.76	0.45	**1.34**	3.6		10	7.5		50	110	40	120	50	
Pentachlorophenol	5	9.12	0.45	**1.35**	6.7	*	10	7.4		40	115	25	130	50	
Benzo(a)anthracene	5	4.33	0.40	**1.21**	3.6		10	8.3		55	110	45	120	50	
Chrysene	5	4.55	0.52	**1.55**	2.9		10	6.4		55	110	45	120	50	
bis(2-ethylhexyl)phthalate	5	3.59	0.27	**0.81**	4.4		10	12.3		40	125	30	140	50	
Butylbenzylphthalate	5	4.26	0.47	**1.40**	3.0		10	7.1		45	115	35	130	50	
Pyrene	5	4.24	0.49	**1.46**	2.9		10	6.8		50	130	35	140	50	
Di-n-octylphthalate	5	2.88	0.19	**0.58**	5.0		10	17.2		35	135	20	155	50	
Indeno(1,2,3-cd)pyrene	5	3.17	0.23	**0.70**	4.5		10	14.2		45	125	30	140	50	
Benzo(b)fluoranthene	5	3.75	0.36	**1.07**	3.5		10	9.3		45	120	35	130	50	
Benzo(k)fluoranthene	5	4.87	0.40	**1.20**	4.1		10	8.3		45	125	30	135	50	
Benzo(g,h,i)perylene	5	3.85	0.37	**1.10**	3.5		10	9.1		40	125	25	135	50	
Benzo(a)pyrene	5	3.90	0.34	**1.01**	3.9		10	9.9		55	110	45	120	50	
Dibenz(a,h)anthracene	5	3.74	0.22	**0.67**	5.6	*	10	14.9		40	125	30	140	50	
Carbazole	5	4.05	0.75	**2.26**	1.8		10	4.4		50	115	35	130	50	
Benzoic Acid										0	125	0	150	50	
3,3'-Dichlorobenzidine										20	110	10	125	50	
N-Nitrosodimethylamine										25	110	10	125	50	
N-Nitrosodiphenylamine										50	110	35	120	50	
1,2-Diphenylhydrazine										55	115	45	120	50	
Benzyl Alcohol										30	110	15	125	50	
Mean Ratio (RD or RQ):					3.8			8.3							
Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 5 and MQL/MDL ratios (RQ) that are less than 3.															
MDL data from two spike levels were combined for this table. QC limits for RPDs are from EM 200-1-3, Appendix I (Feb 2001).															
LCS and MS %R control limits and marginal exceedance (ME) limits are from the DoD QSM, Revision 2 Final, June 2002.															
Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).															
NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.															

TABLE 7
Soil/sediment MDLs, MQLs, and QC Limits for LCS and MS

Semivolatile Organic Compounds (SVOCs) Analysis by GC/MS														
Analytical Management Laboratories														
Matrix: Soil	Preparation Method			3550 & 3541										
	Analytical Method			8270D										
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**			**DoD QSM Control Limits LCS & MS**		**DoD QSM ME Limits LCS & MS**		**RPD**
Compound	Amt.	µg/kg	µg/kg	**µg/kg**	RD =B/A	flag	**µg/kg**	RQ =C/A	flag	Lower	Upper	Lower	Upper	Limit
	µg/kg	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%
Bis(2-Chloroethyl)ether	83	60	4.6	**14.4**	4.2		330	22.9		40	105	25	115	60
Bis(2-chloroisopropyl)ether	83	61	4.7	**14.7**	4.1		330	22.5		20	115	10	130	60
2-Chlorophenol	167	122	6.4	**20.2**	6.1		330	16.3		45	105	35	115	60
1,3-Dichlorobenzene	83	60	2.3	**7.3**	8.3		330	45.5		40	100	30	110	60
1,4-Dichlorobenzene	83	61	2.0	**6.1**	10.0		330	53.9		35	105	25	115	60
1,2-Dichlorobenzene	83	62	2.3	**7.3**	8.4		330	45.0		45	95	35	105	60
Hexachloroethane	83	54	2.2	**6.8**	7.9		330	48.3		35	110	20	120	60
2-Methylphenol (o-Cresol)	83	38	7.2	**22.5**	1.7		330	14.7		40	105	30	115	60
3/4-Methylphenol (m/p-Cresol)	83	37	7.6	**23.9**	1.5		330	13.8		40	105	30	120	60
N-Nitroso-di-n-propylamine	83	44	3.3	**10.5**	4.2		330	31.4		40	115	30	125	60
Phenol	83	67	3.3	**10.2**	6.5		330	32.3		40	100	30	110	60
Bis(2-chloroethoxy)methane	83	59	3.3	**10.3**	5.8		330	32.1		45	110	30	120	60
4-Chloroaniline	167	23	4.0	**12.7**	1.8		330	26.0		10	95	0	110	60
4-Chloro-3-methylphenol	83	51	4.5	**14.3**	3.6		330	23.1		45	115	35	125	60
2,4-Dichlorophenol	83	51	2.8	**8.9**	5.7		330	37.2		45	110	35	120	60
2,4-Dimethylphenol	167	45	7.1	**22.4**	2.0		330	14.7		30	105	20	115	60
Hexachlorobutadiene	83	61	2.8	**8.9**	6.8		330	36.9		40	115	25	130	60
Isophorone	83	75	4.8	**15.2**	4.9		330	21.7		45	110	30	125	60
2-Methylnaphthalene	83	59	1.4	**4.5**	13.0	*	330	73.1		45	105	35	115	60
Naphthalene	167	130	5.7	**18.0**	7.2		330	18.4		40	105	30	120	60
Nitrobenzene	83	63	4.6	**14.5**	4.4		330	22.8		40	115	30	125	60
2-Nitrophenol	83	35	3.8	**11.9**	3.0		330	27.7		40	110	30	120	60
1,2,4-Trichlorobenzene	83	64	2.6	**8.2**	7.8		330	40.1		45	110	30	120	60
Acenaphthene	83	61	2.5	**7.9**	7.6		330	41.6		45	110	35	120	60
Acenaphthylene	83	60	2.7	**8.5**	7.0		330	39.0		45	105	35	115	60
2-Chloronaphthalene	83	65	2.4	**7.5**	8.6		330	44.0		45	105	35	115	60
4-Chlorophenyl-phenylether	83	64	2.0	**6.1**	10.4	*	330	53.8		45	110	35	120	60
Dibenzofuran	83	63	2.6	**8.3**	7.7		330	40.0		50	105	40	110	60
Diethylphthalate	83	68	2.9	**9.0**	7.6		330	36.8		50	115	40	125	60
Dimethylphthalate	83	64	2.8	**8.9**	7.2		330	37.0		50	110	40	120	60
2,4-Dinitrophenol	167	25	2.9	**9.2**	2.7		330	35.8		15	130	10	150	60
2,6-Dinitrotoluene	83	22	3.9	**12.4**	1.8		330	26.7		50	110	35	125	60
2,4-Dinitrotoluene	83	21	3.7	**11.5**	1.9		330	28.8		50	115	35	130	60
Fluorene	83	59	2.6	**8.2**	7.2		330	40.3		50	110	40	115	60
Hexachlorocyclopentadiene	167	104	11.3	**35.5**	2.9		330	9.3		40	115	25	130	60
2-Nitroaniline	83	38	5.9	**18.5**	2.0		330	17.8		45	120	30	130	60
3-Nitroaniline	167	58	7.7	**24.1**	2.4		330	13.7		25	110	15	125	60

TABLE 7 (contd.)
Soil/Sediment MDLs, MQLs, and QC Limits for LCS and MS

Semivolatile Organic Compounds (SVOCs) Analysis by GC/MS															
Analytical Management Laboratories															
Matrix: Soil	Preparation Methods			3550 & 3541											
	Analytical Method			8270D											
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**			**DoD QSM Control Limits LCS & MS**		**DoD QSM ME Limits LCS & MS**		**RPD**
Compound	Amt.	µg/kg	µg/kg	**µg/kg**	RD =B/A	Flag	**µg/kg**	RQ =C/A	flag	Lower	Upper	Lower	Upper	Limit	
	µg/kg	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%	
4-Nitroaniline	167	72	5.3	**16.7**	4.3		330	19.7		35	115	20	125	60	
4-Nitrophenol	83	24	4.8	**14.9**	1.6		330	22.1		15	140	10	160	60	
2,4,6-Trichlorophenol	83	50	3.6	**11.3**	4.4		330	29.2		45	110	30	120	60	
2,4,5-Trichlorophenol	83	49	4.9	**15.4**	3.2		330	21.4		50	110	40	120	60	
Anthracene	83	54	4.1	**12.8**	4.2		330	25.7		55	105	45	115	60	
Phenanthrene	167	144	6.1	**19.3**	7.5		330	17.1		50	110	40	120	60	
4-Bromophenyl-phenylether	83	64	3.3	**10.4**	6.2		330	31.8		45	115	35	13	60	
Di-n-butylphthalate	83	76	4.7	**14.8**	5.1		330	22.3		55	110	45	120	60	
2-Methyl-4,6-dinitrophenol	167	38	4.2	**13.2**	2.8		330	25.0		30	135	10	155	60	
Fluoranthene	83	64	3.9	**12.3**	5.1		330	26.7		55	115	45	125	60	
Hexachlorobenzene	83	71	3.2	**10.0**	7.1		330	33.2		45	120	35	130	60	
Pentachlorophenol	83	42	5.5	**17.4**	2.4		330	19.0		25	120	10	135	60	
Benzo(a)anthracene	167	146	7.0	**21.8**	6.7		330	15.1		50	110	40	120	60	
Chrysene	167	136	6.5	**20.4**	6.7		330	16.2		55	110	45	120	60	
bis(2-ethylhexyl)phthalate	167	160	9.6	**30.2**	5.3		330	10.9		45	125	35	140	60	
Butylbenzylphthalate	83	55	7.0	**22.1**	2.5		330	15.0		50	125	35	135	60	
Pyrene	83	58	5.5	**17.3**	3.3		330	19.1		45	125	35	135	60	
Di-n-octylphthalate	83	56	12.6	**39.6**	1.4		330	8.3		40	130	25	145	60	
Indeno(1,2,3-cd)pyrene	83	88	7.8	**24.5**	3.6		330	13.5		40	120	25	135	60	
Benzo(b)fluoranthene	83	37	2.4	**7.4**	5.0		330	44.3		45	115	35	125	60	
Benzo(k)fluoranthene	83	41	1.8	**5.8**	7.1		330	56.9		45	125	30	135	60	
Benzo(g,h,i)perylene	83	50	4.2	**13.3**	3.8		330	24.9		40	125	25	140	60	
Benzo(a)pyrene	83	38	2.7	**8.4**	4.5		330	39.1		50	110	40	120	60	
Dibenz(a,h)anthracene	83	48	5.8	**18.2**	2.6		330	18.1		40	125	25	140	60	
Carbazole	83	34	5.2	**16.4**	2.1		330	20.2		45	115	30	130	60	
Benzoic Acid										0	110	0	130	60	
3,3'-Dichlorobenzidine										10	130	0	145	60	
N-Nitrosodimethylamine										20	115	10	130	60	
N-Nitrosodiphenylamine										50	115	40	125	60	
1,2-Diphenylhydrazine										55	115	45	120	60	
Benzyl Alcohol										20	125	10	140	60	
Mean Ratio (RD or RQ):					5.1			28.8							
Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 10 and MQL/MDL ratios (RQ) that are less than 3.															
MDL studies were performed at two spike levels and the data combined as shown above. QC limits for RPDs are from EM 200-1-3.															
LCS and MS %R control limits and marginal exceedance (ME) limits are from the DoD QSM, Revision 2 Final, June 2002.															
Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).															
NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.															

Table 8
List of Appendices

Appendix Number	Appendix Topic
1	Chromatographic Conditions (Instrument Printouts) DFTPP Tune Summary (Form-5 equivalents)
2	Initial Calibration (ICAL) Summary (Form-6 equivalents) Calibration Verification (ICV and CCV) Summary (Form-7 equivalents)
3	Instrument Printouts -Quantitation Reports and Sample Chromatograms
4	Instrument Printouts -Spectral Identification
5	Instrument Printouts -Library Search
6	Method Detection Limits (MDLs) Study Results
7	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
8	USACE Method Quality Objectives for Method 8270C AFCEE MQOs for Method 8270C DoD QSM MQOs and Limits for percent recoveries
9	Internal Standard Area and RT Reports (Form-8 equivalents)
10	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
11	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
12	QC Association Forms (Form-4 equivalents) Surrogate Recovery Summaries (Form-2 equivalents) Sample Reports (Form-1 equivalents) - Soil and Water
13	Sample Preparation Log Instrument Analysis Log

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. O-DW-SOCs-RC

Safe Drinking Water Act (SDWA) Semivolatile Organic Compounds (SOCs) by Solid Phase Extraction (SPE) and Instrumental Analysis By Gas Chromatography/Mass Spectrometry (GC/MS) (EPA 525.2)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Safe Drinking Water Act (SDWA) Semivolatile Organic Compounds (SOCs) by Solid Phase Extraction (SPE) and Instrumental Analysis By Gas Chromatography/Mass Spectrometry (GC/MS) (EPA 525.2)

1.0 SCOPE AND APPLICATION

1.1 This Analytical Management Laboratories (AML) standard operating procedure (SOP) is based on EPA Method 525.2. Words and acronyms such as method, procedure, and SOP are used interchangeably in this document. This SOP is used to determine semivolatile organic compounds in drinking water and groundwater samples only.

1.2 This SOP can be used to quantitate most semivolatile organic compounds (SOCs) that have an affinity for the C-18 sorbent, which is used to extract them from the water samples and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with DB5. ***Table 1*** lists compounds that are regulated by the Safe Drinking Water Act (SDWA), their regulatory limit, gas chromatographic retention times and the ions used for identification and quantitation. This SOP includes detailed procedures for the analysis of the SDWA-SOCs. Table 1 also includes internal standards and surrogates that are employed for this method.

1.3 ***Tables 2 to 5*** lists compound classes that can also be determined using this SOP. Attempting to determine all of the above analytes is not practical and not necessary in most cases. If all the analytes must be determined, multiple calibration mixtures will be required.

1.4 ***Table 4*** describes organochlorine pesticides and PCBs that can be determined using this method. The laboratory will rely on other, more sensitive methods (e.g. 508, and 508.1) for their determination.

1.5 The method detection limits (MDLs) and method quantitation limits (MQLs) for each individual compound is instrument dependent and highly matrix dependent. The instrument employed at AM Laboratories is a standard Agilent (formerly HP) quadrupole 5973 mass selective detector (MSD). The concentration calibration range demonstrated in this method is 0.5 to 10-µg/L for most analytes. See ***Table 10*** for MDLs and MQLs that are generally achievable for the SOCs.

1.6 This SOP uses gas chromatography/mass spectrometry (GC/MS) procedures. This SOP is restricted to experienced analysts in the use of GC/MS theories and skilled in the interpretation of mass spectra and their use as a quantitative tool.

1.7 This SOP differs significantly from the SOP for SVOCs based on 625 and 8270C for wastewater, groundwater and soil samples. For this SOP, the internal standards are added to the samples at the same time as surrogates, prior to sample extraction.

2.0 SUMMARY OF METHOD

2.1 Organic compound analytes, internal standards, and surrogates are extracted from a water sample by passing 1-L of sample water through a cartridge or disk containing a solid matrix with a chemically bonded C_{18} organic phase (liquid-solid extraction, LSE). The

organic compounds are eluted from the LSE cartridge or disk with small quantities of ethyl acetate followed by methylene chloride, and this extract is concentrated further by evaporation of some of the solvent.

2.2 The sample components are separated, identified, and measured by injecting an aliquot of the concentrated extract into a high-resolution fused silica capillary column of a gas chromatography/mass spectrometry (GC/MS) system. Compounds eluting from the GC column are identified by comparing their measured mass spectra and retention times to reference spectra and retention times in a database.

2.3 Reference spectra and retention times for analytes are obtained by the measurement of calibration standards under the same conditions used for samples. The concentration of each identified component is measured by relating the MS response of the quantitation ion produced by that compound to the MS response of the quantitation ion produced by a compound that is used as an internal standard.

2.4 Surrogate analytes, whose concentrations are known in every sample, are measured with the same internal standard calibration procedure.

2.5 Spiked samples are used to verify the chosen extraction procedure for each matrix.

3.0 INTERFERENCES

3.1 Interferences can be caused by a number of sources in the extraction and analyses stages. During analysis, major contaminant sources are reagents and liquid-solid extraction devices. Quality control measures, such as method blanks, solvent blanks and matrix spikes can be used to determine the interference.

3.2 Interfering contamination may occur when a sample containing low concentrations of compounds is analyzed immediately after a sample containing relatively high concentrations of compounds. Syringes and splitless injection port liners must be cleaned carefully or replaced as needed. After analysis of a sample containing high concentrations of compounds, a laboratory reagent blank should be analyzed to ensure that accurate values are obtained for the next sample.

3.3 To reduce carryover, the sample syringe must be rinsed out between samples with solvent.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph - A Hewlett Packard 6890 Series II GC is used. The system is complete with a temperature-programmable GC for splitless injection. The system includes all accessories, including syringes, analytical columns and gasses. The column flow rate remains constant throughout temperature program operation. The capillary column is directly coupled to the source.

4.2 Gas chromatographic column - A 30 meter x 0.25-mm ID capillary column coated with DB-5 (J&W Scientific) is employed. The film thickness is 0.25 um. An equivalent column may be used.

4.3 Mass Spectrometer - A Hewlett Packard 5973 Mass Selective Detector is used. It is capable of scanning from 35 to 800 AMU every 1-second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The spectrometer should produce a mass spectrum that meets all criteria in Table 8 when an injection of

approximately 5-ng of DFTPP is introduced into the GC. An average spectrum across the DFTPP GC peak may be used to test instrument performance. GC/MS interface is directly coupled by inserting the column into the mass spectrometer.

4.4 Data system - Hewlett Packard's Enviroquant is used to allow continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the GC program. The software allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). The software also allows integrating the abundances in any EICP between specified times or scan-number limits.

4.5 Balance - Analytical, 0.0001 grams and top loading, 0.1 grams.

4.6 Heated water bath

4.7 SPE extraction manifold (Supelco) with 12-positions for sample extraction, vacuum control and reservoir for spent water sample collection (Kontes Ultraware).

4.8 Vacuum pump (Gast Model 2Z866).

4.9 Liquid-Solid Extraction (LSE) Cartridges - Cartridges are inert non-leaching plastic, for example polypropylene, or glass, and must not contain plasticizers, such as phthalate esters or adipates, that leach into the ethyl acetate and methylene chloride eluant. The cartridges are packed with about 1 gram of silica, or other inert inorganic support, whose surface is modified by chemically bonded octadecyl (C_{18}) groups. The packing must have a narrow size distribution and must not leach organic compounds into the eluting solvent. One liter of water should pass through the cartridge in about two hours with the assistance of a slight vacuum of about 13-cm (5 in.) of mercury. The laboratory uses Discovery DSC-18 solid phase extraction (SPE) cartridges (6-mL tubes, 500-mg) with C-18 phase from Supelco (Catalogue # 52604-U).

4.10 **NOTE:** All glassware specified in this SOP should be meticulously cleaned. This may be accomplished by washing with detergent and water, rinsing with water, distilled water, or solvents, air-drying, and heating (where appropriate) in a muffle furnace. Volumetric glassware should never be heated to the temperatures obtained in a muffle furnace.

4.11 Microsyringes - 10, 25, 100, 250, 500 and 1000 uLs.

4.12 Disposable pipettes - Pasteur

4.13 Test tubes - 5 ml to 8 ml, with Teflon lined screw caps.

4.14 Microsyringes - 10-, 50-, 100-, 500-, and 1000-uL.

4.15 Glass wool

4.16 Stainless steel table spoons

4.17 Drying Column -- The drying tube should contain about 5-7 g of anhydrous sodium sulfate to prohibit residual water from contaminating the extract. Any small tube may be used, such as a syringe barrel, a glass dropper, etc. as long as no sodium sulfate passes through the column into the extract.

5.0 REAGENTS

5.1 It is assumed that all inorganic/organic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where available. Any other reagents must demonstrate high purity without lessening the accuracy of the determination.

5.2 Organic free reagent water - All references to water in this method will be assumed to be organic free reagent water, except when referring to field samples. Use Type 1 reagent water to prepare MB and LCS samples. See AML SOP on reagent water for more details on water quality.

5.3 Acids

5.3.1 Sulfuric Acid (1:1 v/v) Solution - Slowly add 50-mL of concentrated sulfuric acid to 50-mL of water.

5.3.2 Hydrochloric Acid (6N) Solution - Slowly add 50-mL of concentrated Hydrochloric acid to 50-mL of water (1:1 v/v).

5.4 Solvents

5.4.1 Acetone – GC or pesticide grade

5.4.2 Hexane – GC or pesticide grade

5.4.3 Methylene chloride - GC grade

5.4.4 Ethyl acetate - GC or pesticide grade

5.4.5 Toluene - GC or pesticide grade

5.4.6 Methanol - GC or pesticide grade

5.5 Stock Standards - **NOTE:** For neat standards with compound purity that is confirmed by the supplier at >96%, the weighed amount can be used without correction to calculate the concentration of the solution. Do not put every method analyte in a single primary dilution standard because chromatographic separation will be extremely difficult, if not impossible. Two or three primary dilution standards would be more appropriate. The recommended solvent for these standards is acetone or ethyl acetate. Aliquots of each of the stock standard solutions are combined to produce the primary dilution in which the concentration of the analytes is at least equal to the concentration of the most concentrated calibration solution. Store the primary dilution standard solution in an amber vial in a dark cool place, and check frequently for signs of degradation or evaporation, especially just before preparing calibration solutions. Stock standard solutions expire one year from the date of preparation. Replace sooner if signs of degradation are observed.

5.5.1 Stock Standard Solutions - The primary list of target compounds for this SOP is shown in Table 1. Stock standards for the primary list are purchased from Ultra Scientific as shown below:

5.5.1.1 Alachlor (Catalog number EPA-1068) at a concentration of 5000-ug/mL.

5.5.1.2 Atrazine (Catalog number EPA-1176A) at a concentration of 1000-ug/mL.

5.5.1.3 Simazine (Catalog number PP-530A) at a concentration of 100-ug/mL.

5.5.1.4 Compounds #3 to 8 in Table 1 (Catalog number SVM-500-1) at a concentration of 500-ug/mL each.

5.6 Fortification Solution of Internal Standards and Surrogates - One solution that contains the three internal standards and the four surrogates is used. Purchase the mix from Ultra (Catalogue #ISM-510) that contains the internal standards (acenaphthene-D_{10}, phenanthrene-D_{10}, and chrysene-D_{12} in acetone) at a concentration of 500-µg/mL of each and the surrogate compounds (1,3-dimethyl-2- nitrobenzene, perylene-D_{12}, triphenylphosphate, and pyrene-D_{10} in acetone) at a concentration of 500-µg/mL.

5.6.1 This solution is used in the preparation of the calibration solutions. Dilute a portion of this solution by 10 to a concentration of 50-µg/mL and use this solution to fortify the

actual water samples. A 100-µL aliquot of this 50-µg/mL solution added to 1-L of water gives a concentration of 5-µg/L of each internal standard or surrogate.

5.6.2 Store these solutions in an amber vial in a dark cool place.

5.7 Fortification Solution for Recovery Standard - Purchase the standard solution from Ultra (Catalogue #ATS-160-1) that contains p-terphenyl-D_{14} at a concentration of 500-µg/mL in methylene chloride or ethyl acetate. An aliquot (10-µL) of this solution should be added to each extract (1-mL) to check on the recovery of the internal standards in the extraction process. The final concentration of the recovery standard is 5-µg/mL in the extract.

5.8 Initial Calibration (ICAL) Solutions (see Table 6) - **NOTE:** Procedures that are similar to those described below are used to prepare ICAL standards for other parameters shown in Tables 2 to 4. The ICAL working standards are prepared from the Stock Standard Solutions (Section 5.5, 5.6 and 5.7) as described in Table 6. The ICAL standards are prepared at six concentration levels for each parameter of interest: 0.2-µg/mL, 1.0-µg/mL, 2.5-µg/mL, 5-µg/mL, 10-µg/mL, and 20-µg/mL for the target analytes. The ICAL solutions also contain the internal, surrogate and recovery standards at fixed (5-µg/mL) concentrations. When six levels of standards are prepared, the analyst may use all six points or drop one point (preferably the lowest point for analytes such as pentachlorophenol), if sensitivity problem and/or curvature is suspected. Prepare the ICAL standards as described below using ethyl acetate as the solvent.

5.8.1 Prepare an intermediate stock standard that contains all the nine Target compounds at 40-µg/mL by diluting 160-µL of the mixed standard (Section 5.5.1.4), 16-µL of the Alachlor standard (Section 5.5.1.1), 80-µL of the Atrazine standard (Section 5.5.1.2), and 800-µL of the Simazine standard (Section 5.5.1.3) to 2-mL with ethyl acetate. This standard is fortified with internal standard/surrogate standard (IS/SS, Section 5.6) and the recovery standard (RS, Section 5.7) to prepare ICAL solutions for a final volume of 1-mL for each ICAL solution. Scale up as needed to prepare additional quantities.

5.8.2 Prepare ICAL-1 by diluting 5-µL of the mixed standard (Section 5.8.1) to 1-mL with ethyl acetate. Spike it with 10-µL of the IS/SS standard and 10-µL of the RS standard.

5.8.3 Prepare ICAL-2 by diluting 25-µL of the mixed standard (Section 5.8.1) to 1-mL with ethyl acetate. Spike it with 10-µL of the IS/SS standard and 10-µL of the RS standard.

5.8.4 Prepare ICAL-3 by diluting 62.5-µL of the mixed standard (Section 5.8.1) to 1-mL with ethyl acetate. Spike it with 10-µL of the IS/SS standard and 10-µL of the RS standard.

5.8.5 Prepare ICAL-4 by diluting 125-µL of the mixed standard (Section 5.8.1) to 1-mL with ethyl acetate. Spike it with 10-µL of the IS/SS standard and 10-µL of the RS standard.

5.8.6 Prepare ICAL-5 by diluting 250-µL of the mixed standard (Section 5.8.1) to 1-mL with ethyl acetate. Spike it with 10-µL of the IS/SS standard and 10-µL of the RS standard.

5.8.7 Prepare ICAL-6 by diluting 500-µL of the mixed standard (Section 5.8.1) to 1-mL with ethyl acetate. Spike it with 10-µL of the IS/SS standard and 10-µL of the RS standard.

5.8.8 **NOTE:** Prepare the ICAL standards in GC injection vials. After injection, the standards may be removed from the GC auto sampler, re-crimped with new septa, and stored in a freezer for re-use up to three months.

5.9 Initial Calibration Verification (ICV) Solution - This mid-level standard that is identical in composition to the ICAL-4 standard (Section 5.8.5) is used to verify the ICAL constructed with standards described above (Section 5.8). It is prepared from the IS/SS and RS standards that are of the same source and the target analytes that are from a different source (Supelco). Table 7 for a list of Supelco standards that are used to prepare ICV.

5.10 Continuing Calibration Verification (CCV) Solution - This is identical to the ICAL-4 standard (Section 5.8.5). A second aliquot of the ICAL-4 is prepared and labeled CCV.

5.11 Instrument Performance Check Solution (IPC) - This semivolatiles GC/MS tuning standard solution may contain one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of method criteria. This solution, which contains DFTPP, 4,4'-DDT, PCP, & Benzidine, each at a concentration of 1000-ug/mL is purchased from Ultra (cat # GCM-150). Prepare a 50-ug/ml standard by diluting 1.0-ml of the Ultra Scientific standard to 20-ml with methylene chloride. The tuning standard solution expires one year from the date of preparation. Replace sooner if signs of degradation are observed.

5.12 LCS spiking solution is prepared from the same source as the ICAL standard. Prepare spiking solution at a concentration of 5-µg/mL by diluting 500-µL of the 40-µg/mL mixed standard (Section 5.8.1) to 4.0-mL with methanol. Spike 1.0-mL of this solution 10 1.0-L of reagent water to prepare LCS and LCSD samples. The spiking solution expires in ninety days from the date of preparation. Replace sooner if signs of degradation are observed.

5.13 Matrix Spike spiking solution is identical to the LCS spiking solution (Section 5.12). Spike 1-mL of the solution to 1.0-L of the water designated for MS and MSD analyses.

5.14 Sodium Hydroxide Solution (10N) - Dissolve 40 grams of sodium hydroxide solution into 100 mLs of reagent water.

5.15 Sodium sulfate - (granular, anhydrous), Na_2SO_4, Soxhlet extracted with methylene chloride for a minimum of four hours or heated to 400°C for two hours in a muffle furnace.

5.16 Reducing Agent - Sodium sulfite, anhydrous. Sodium thiosulfate is not recommended as it may produce a residue of elemental sulfur that can interfere with some analytes.

5.17 Buffer for conditioning SPE tubes - Prepare 25-mM KH_2PO_4 (Sigma, catalogue # P-0662, F.W = 136.1) by dissolving 13.6-g in 400-mL of reagent water.

5.18 Helium Carrier Gas - Ultra High Purity (as contaminant free as possible).

6.0 SAMPLE PRESERVATION HANDLING & STORAGE

6.1 One liter amber glass bottles with Teflon lined screw caps may be used for collecting aqueous samples in accordance with EPA 525.2.

6.1.1 Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Add 1-mL of 6N hydrochloric acid (Section 5.3.2) to each bottle. Provide 2 bottles for each water sample to be collected.

6.1.2 Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples.

6.1.3 See project specific field sampling plan (FSP) for sample collection and preservation instructions. No preservatives are required for groundwater samples.

6.1.4 Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.

6.2 Sample Collection Procedures. These are reproduced from EPA 525.2

6.2.1 When sampling from a water tap, open the tap and allow the system to flush until the water temperature has stabilized (usually about two minutes). Adjust the flow to about 500-mL/min and collect samples from the flowing stream. Keep samples sealed from collection time until analysis.

6.2.2 When sampling from an open body of water, fill the sample container with water from a representative area. Sampling equipment, including automatic samplers, must be free of plastic tubing, gaskets, and other parts that may leach interfering analytes into the water sample. Automatic samplers that composite samples over time should use refrigerated glass sample containers if possible.

6.2.3 Chlorinated drinking water samples will need special attention. See Section 6.3 for details.

6.2.4 Samples should be iced or refrigerated at 4°C and kept in the dark from the time of collection until extraction.

6.3 Sample Dechlorination and Preservation - Residual chlorine should be reduced at the sampling site by addition of 40-50 mg of sodium sulfite (this may be added as a solid with stirring or shaking until dissolved) to each water sample. It is very important that the sample be dechlorinated prior to adding acid to lower the pH of the sample. Adding sodium sulfite and HCl to the sample bottles prior to shipping to the sampling site is not permitted.

6.4 Sample Preservation - Hydrochloric acid should be used at the sampling site to retard the microbiological degradation of some analytes in water. The sample pH is adjusted to <2 with 6 N hydrochloric acid. This is the same pH used in the extraction, and is required to support the recovery of acidic compounds like pentachlorophenol.

6.5 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Analytical data generated outside of the specified holding times must be considered to be minimum values only.

6.6 Holding Times - The following statement is reproduced from EPA Method 525.2: *"Results of the time/storage study of all method analytes showed that all but six compounds are stable for 14 days in water samples when the samples are dechlorinated, preserved, and stored as described in Section 8.2. Therefore, samples must be extracted within 14 days and the extracts analyzed within 30 days of sample collection".* The standard holding times (from 40CFR Part 136) for extraction of aqueous samples is 7-days from the date of collection. Sample extracts should be analyzed within 40-days from the date of extraction. The laboratory should follow these guidelines and make every effort to meet each criterion to the extent possible.

7.0 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control contain a number of regulated compounds at high concentrations. The toxicity of these compounds present in such complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

7.3 Some method analytes have been tentatively classified as known or suspected human or mammalian carcinogens. Pure standard materials and stock standard solutions of these compounds should be handled with suitable protection to skin, eyes, etc.

7.4 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.5 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.6 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Aqueous samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters after the solvent(s) have been allowed to evaporate in a hood.

9.0 SAMPLE EXTRACTION PROCEDURE

9.1 Preparation of Water Samples

9.1.1 Samples should be at room temperature. Allow them to warm up to room temperature. The pH of the water samples should be less than 2. Measure using a pH paper and record it in the logbook. This is accomplished by dipping the tip of a Pasteur pipette into the water and placing the droplets of water on the pH paper. Never place the pH paper into the water sample. Adjust pH to <2 if necessary with 50 % H_2SO_4.

9.1.2 Using a black marker, mark the meniscus of the water in the sample container.

9.1.3 Prepare MB, LCS, LCSD, MS, and MSD samples as described previously.

9.1.4 When the extraction is complete (see Section 9.2) measure the extracted volume for each sample as described below.

9.1.5 Add tap water to the empty sample container up to the marked meniscus. Pour the water into a 2-liter graduated cylinder. Record this measured volume in the logbook.

9.2 Sample Extraction

9.2.1 Add 100-µL of the internal standard/surrogate solution (see Section 5.6.1) to all samples (includes QC samples such as MB, LCS, LCSD, MS and MSD).

9.2.2 Perform extractions using Supelco SPE vacuum manifold (Section 4.7). Condition the Discovery SPE tube (see Section 4.9) to activate the sorbent before extracting the samples.

9.2.2.1 Clean each tube by eluting with 5-mL of ethyl acetate and 5-mL of methylene chloride. Condition the tubes with three tube volumes (10-mL) of methanol, followed by 5-mL of reagent water. The final rinse is performed with 5-mL of 25-mM KH_2PO_4 (Section 5.17) for maximizing pentachlorophenol recoveries. A thin film of buffer should remain on the sorbent and the tube should not dry up anytime during the conditioning procedure. Disconnect vacuum source (Section 4.8) before setting up sample extraction.

9.2.3 Dip one end of the sample drain tube into the bottom of the sample bottle and snap the other end into the SPE cartridge. Adjust the flow rate of the sample through the SPE cartridge to less than 10-mL/minute by adjusting the vacuum controller for each sample in the manifold.

9.2.4 After all the samples have passed through the cartridge, but before the SPE tube goes dry, remove the connecting tube from the cartridge. Rinse the bottles with 5-mL reagent water and allow them to go through the tube. Repeat the rinse step. (**NOTE:** A wash step with 2-mL of 5% methanol in reagent water may be necessary to remove weakly retained materials. The effect of wash step on target compound recoveries should be established by performing them on LCS and LCSD.)

9.2.5 Elute the target compounds with 2.0-mL ethyl acetate, followed by two 2-mL methanol. Pulse the vacuum pump to start the flow, then let the solvent drip through under gravity alone at a flow rate of approximately 1-mL/minute. Collect the extract in clean 10-mL screw-cap tubes.

9.2.6 Concentrate the extract by nitrogen blow-down to 1.0 mL. Spike each extract with 10-µL of the recovery standard (Section 5.7) solution. The final concentration of the recovery standard is 5-µg/mL in the extract. Proceed to analyze by GC/MS.

10.0 GC/MS CALIBRATION PROCEDURE

10.1 Chromatographic conditions - Shown below (see Appendix 1 for details):

Injection port: 250°C

Transfer line temperature: 300°C
Split ratio: splitless during injection (2-minutes) and 5:1 thereafter
Split flow: 6-mL/min.
Carrier gas flow rate: 15 mL/min.
Initial temperature: 50°C, hold for 2-minutes
Temperature program: 6°C /min
Final temperature: 300°C, hold for 2-minutes

10.2 Initial calibration - the MS operation conditions

Mass range: 40 to 600-AMU
Scan rate: 1-sec/scan
Source temperature: 230°C

10.3 Mass Spectrometer Tune and GC Column Performance

10.3.1 The mass spectrometer is tuned (See Appendix 1 for details on documentation) to meet the criteria in ***TABLE 8,*** *every 12 hours*, for the injection of 5-ng of DFTPP, 4,4'-DDT, PCP, & Benzidine or instrument performance check (IPC) solution (Section 5.11). Inject the tuning standard and using the mass abundances determine if the criteria are met for DFTPP as stated in ***TABLE 8***. Background subtraction should be straightforward, only to eliminate column bleed or instrument background ions. If it is not met, retune the mass spectrometer and reanalyze the DFTPP standard. Analyses of the initial calibration cannot be performed until the criteria are met.

10.3.2 The GC/MS tuning standard should also be used to assess GC column performance and injection port inertness. DDT breakdown to DDD and DDE should not exceed 20%. Corrective action such as injection port maintenance must be performed when degradation is excessive. The calculation for % breakdown is as follows:

$$\%\ \text{breakdown (DDT)} = \frac{\text{Peak area of (DDE + DDD)} * 100}{\text{Peak area of (DDT+DDE +DDD)}}$$

10.3.3 Measure peak tailing as shown in Figure 1. Quantitative peak tailing factors of 3.0 and 5.0 are recommended for benzidine and pentachlorophenol respectively. Check also the instrument response for these compounds. Clean the injection port and/or break off the first 10- to 25-cm of the capillary column as needed to restore GC performance.

10.4 Initial Calibration (ICAL) - Once the criteria in section 7.3 have passed, analysis to set up the initial calibration can be performed. Inject the six initial calibration standards (Section 5.9) starting with the mid-level standard (ICAL-4). The ICAL-1 is run for projects requiring lower MQLs and to obtain an MQL equal to the EPA maximum contaminant level (MCL). The ICAL-2 is normally used to set MQLs for most other compounds. The calibration curve may be run from high to low with the exception of ICAL-4, which is run first.

10.4.1 Summarize the initial calibration (see Appendix 2 for details) data in Form-6 equivalents. After the completion of ICAL-4 standard, ensure that all compounds have been appropriately identified.

10.4.2 A six-point calibration is not required, although it is preferred, when possible. Use all six points or drop one of the points (either the lowest or the highest, but not any of the middle points) if curvature is a problem for some of the compounds.

10.5 Tabulate the area response of the characteristic ions against concentration for each compound and each standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard that corresponds to the SVOC compounds is shown in ***TABLE 1***. The RF is calculated as shown below:

$$RF = (A_x C_{is}) / (A_{is} C_x)$$

where:
Ax = Area of the characteristic ion for the compound being measured
Ais = Area of the characteristic ion for the specific internal standard
Cis = Concentration of the specific internal standard
Cx = Concentration of the compound being measured

10.6 The average RF must be calculated and recorded for each compound using five to seven RF values calculated from the initial calibration analysis.

10.7 Using the RFs from the initial calibration, calculate and record the percent relative standard deviation (%RSD) for all the compounds. The percent RSD is calculated as follows:

$$\%RSD = ((SD)/(AVER\ RF))*100\%$$

where:
AVE RF = mean of the 5or 6 initial RFs for a compound
SD = standard deviation of the 5or 6 initial RFs for a compound.

The percent relative standard deviation should be less than 30% for each target analyte.

10.8 If a %RSD greater than 30% is measured for any compound, then corrective action to eliminate a system leak and/or column active sites is required before reattempting the calibration. The calibration curve must be re-analyzed if the RSD for any compound remains above 30%.

10.9 Using a linear or non-linear regression method is also an option for initial calibration. The correlation coefficient for linear regression should be >0.990. Verify the initial calibration with a second source initial calibration verification solution (ICV), prepared as described in Section 5.. The observed results should be within ±30% of the true values for each target compound for the ICAL to be acceptable. Document ICV results in a manner that is similar to the CCV (Section 10.12).

10.10 If the above criteria are met, the analysis for analytical samples can begin.

10.11 Tuning verification - Prior to the analysis of samples outside the 12-hour time limit of the initial calibration, the criteria for the tuning and calibration should be met. Verify tuning by analyzing an aliquot of the instrument performance check (IPC) solution

(Section 5.11) must be met. The criteria described in section 10.3 should be met very 12-hours.

10.12 Continuing Calibration verification (CCV) – The initial calibration curve for each compound of interest must be checked and verified once every 12 hours with a continuing calibration verification (CCV) standard (Section 5.10). Calibration verification is not necessary for samples analyzed immediately after ICAL provided they are within 12-hours of the tune standard analyzed with ICAL.

10.12.1 Analyze the CCV standard, which is identical to the ICAL-4 standard. Summarize CCV in Form-7 equivalents (see Appendix 3 for details).

10.12.2 Tabulate the internal standard (IS) areas in Form-8 equivalents (see Appendix 9 for details). The IS areas for the CCV should be within ±50% of the measured IS area for the ICAL-4 standard. If a previous CCV was analyzed, the IS areas for the current CCV should be within ±30% of the measured IS area for the previous CCV standard.

10.12.3 Calculate either the percent difference (%D) (for response factors) or percent drift (%D) (for linear regression) for the CCCs using one of the following equations:

$$\%D = \%\text{Difference} = ((RF_v - RF_m) * 100) / (RF_m)$$

$$\%D = \%\text{ drift} = ((C_v - C_m) * 100) / (C_m)$$

where:

RF_m = Average or mean response factor from ICAL
RF_v = Observed RF for the CALV standard
C_m = True concentration of the CALV standard.
C_v = Measured concentration of the CALV standard using selected quantitation method (e.g. linear regression).

10.13 If the %D for each compound is less than 30%, the initial calibration is assumed to be valid. If the criterion is not met (i.e., >30%), for any one of the compound, corrective action should be taken. If the source of the problem cannot be detected, then a new five/six point calibration must be generated. This criterion must be met before any quantitative analysis can begin. Not more than 10% of the compounds can miss the %D check. If an analyte that missed the CCV is detected in the samples, a new CCV may be performed after the analysis of the samples to check compliance. Remedial actions that may be performed to restore calibration include the following:

10.13.1 Major maintenance such as cleaning an ion source, cleaning quadrupole rods, replacing filament assemblies, etc. require returning to the initial calibration step.

10.13.2 Check and adjust GC and/or MS operating conditions; check the MS resolution, and calibrate the mass scale.

10.13.3 Clean or replace the splitless injection liner; silanize a new injection liner.

10.13.4 Flush the GC column with solvent according to manufacturer's instructions.

10.13.5 Break off a short portion (about 1 M) of the column from the end near the injector; or replace GC column. This action will cause a change in retention times.

10.13.6 Prepare fresh CAL solutions, and repeat the initial calibration step.

10.13.7 Clean the MS ion source and rods (if a quadrupole).

10.13.8 Replace any components that allow analytes to come into contact with hot metal surfaces.

10.13.9 Replace the MS electron multiplier, or any other faulty components.

11.0 ANALYSIS OF EXTRACTED SAMPLES

11.1 Once the samples have been extracted and all system performance checks have been established as stated in the previous sections, sample extracts can be analyzed. The sample extracts and associated quality control sample (MB, LCS, LCSD, MS, and MSD) extracts are analyzed in the same manner as calibration standards. Document analysis on instrument logs (see Appendix 13 for details).

11.2 If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, reanalyze sample extracts after appropriate dilutions. Additional internal standard must be added to the diluted extract to maintain the required 5-ng/uL level for each internal standard in the extract that is injected. The diluted extract must be reanalyzed.

12.0 DATA INTERPRETATION

12.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum with characteristic ions in reference mass spectrum. The reference mass spectra are established by generating them from the analyses performed during initial calibration. The characteristic ions are defined to be the three ions of the greatest relative intensity or any ions over 30% relative intensity if less then three such ions occur in the reference spectra. Compounds should be identified as present when the criteria below are met.

12.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

12.1.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

12.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

12.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different retention times.

12.1.5 Identification can become difficult when more than one analyte elutes chromatographically causing overlapping of spectra. It is still possible to identify a compound by closely examining the EICP of the appropriate ions. Each analyte spectrum will contain extraneous ions contributed by the co-eluting compounds.

12.2 A library search may be necessary when components are present that are not identified as target analytes. Guidelines for making tentative identification (See also Appendix 4 for details on documentation) are:

12.2.1 Relative intensities of major ions in the reference spectrum should be present in the sample spectrum.

12.2.2 The relative intensities of the major ions should agree within ± 20%.

12.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.4 Ions present in the sample but not in the reference should be reviewed for possible background contamination presence of coeluting compounds.

12.2.5 Ions present in the reference but not in the sample should be reviewed for possible subtraction from sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create discrepancies.

13.0 QUANTITATIVE ANALYSIS

13.1 The internal standard technique is used to determine the concentration of an identified target compound. For water samples the calculation is as follows:

$$\text{Concentration (ug/L)} = \frac{(A_X)\,(Q_{IS})}{(A_{IS})\,(RF_m)\,(V_s)}$$

where:

A_X = Area of characteristic ion for compound being measured
Q_{IS} = Amount of internal standard area added to the sample (µg)
A_{IS} = Area of characteristic ion for the internal standard
RF_m = Mean response factor for compound being measured from ICAL
V_s = Volume of water (L) extracted, taking into account any dilutions made.

13.2 Document sample data (see Appendix 4 for details) in quantitation reports, chromatograms, and spectra for compounds identified in the sample. Prepare Form-1 (see Appendix 5 for details) for client reports.

13.3 Sometimes it is necessary to give an estimated quantitation value for a non-target analytes or tentatively identified compounds (TICs). The equations described above can be used to calculate sample amounts, substituting 1.0 for the RF_m and using the total ion areas in the chromatograms for the unknown and the nearest IS for the unknown. The result for TICs should be identified as an "estimated value only" by following the value with a "J" code. The report should also identify which IS was used to determine the concentration.

14.0 QUALITY CONTROL

Analytical data, which includes calibration data, sample preparation logs, sample data and quality control data should be gathered prior to review. Review checklist (See Appendix 7 for details) should be used to document verification of calculations and adherence to method

procedures. Exceptions should be documented. Samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions should be documented on these checklists.

14.1 Perform method detection limit (MDL) studies using sample spiked close to the estimated MDL (approximately 0.5-µg/L). See EPA 525.2 for guidance on spiking levels and the AML SOP for MDL determination. MDL data are summarized in Table 10. For details on MDL studies see Appendix 6.

14.2 Perform an initial demonstration of capabilities by spiking reagent water with target analytes at 5-µg/L and performing all the steps that are normally performed for environmental samples. Perform the spike recovery studies in quadruplicate. The recoveries for target compounds should be within ±30% of the true value (%R within 70 to 130%). See AML SOP on MDL and initial demonstration for details.

14.3 Analyze a method blank (MB) and spiked blanks (LCS and LCSD) and spiked samples (MS and MSD) with each batch of samples.

14.4 Summarize sample results using sample result Form 1 equivalents (See Appendix 5). Use the same format for all analyses including QC samples such as MB, LCS, LCSD, MS, and MSD. The MDLs, RLs (when applicable), and MQLs applicable to the method are shown in Table 10. Report positive results greater than the MDL and/or the RL but less than the PQL as estimated with a "J" flag. Report positive results that are greater than the upper calibration limit as estimated with an "E" flag.

14.5 Evaluate sample data as shown in the Data Verification section and apply additional qualifiers as needed. Document them in the checklists (see Appendix 7 for details) and in the case narrative provided to clients. Report sample results to the client in reports shown in Form-1 equivalents (see Appendix 5 for details).

14.6 Summarize QC results for LCS/LCSD in Form-1. Summarize recoveries and RPDs for duplicates in Form-3 equivalents (see Appendix 10 for details). Evaluate them as shown in the Data Verification section.

14.7 Summarize QC results for MS/MSD in Form-1. Summarize recoveries and RPDs for duplicates in Form-3 equivalents (see Appendix 11 for details). Evaluate them as shown in the Data Verification section.

14.8 Calculate surrogate recoveries in reference to the internal standards. Calculate internal standard recoveries in reference to recovery standards. Summarize internal standard and surrogate recoveries in Form-2 equivalents (see Appendix 12 for details). Evaluate them as shown in the Data Verification section.

14.9 Summarize method blank (MB) results in the same manner as sample results (see Appendix 5 for details). Summarize MB or QC association in Form-4 equivalents (see Appendix 13 for details). Include sample extraction sheets and copies of the instrument log in project folders. Evaluate them as shown in the Data Verification section.

14.10 Report internal standard areas and retention times in samples in Form-8 equivalents (see Appendix 9 for details).

15 DATA VERIFICATION AND VALIDATION - This section will provide guidance when evaluating and validating data generated by this SOP. It provides procedures to assess if data

produced is valid and legally defensible. Document review and comments on the checklist (see Appendix 7 for details). Verify that sample preparation logs are complete and the instrument run logs are available for review. Anomalies and exceptions should be documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on these checklists. USACE projects may have special requirements that will require extra attention. A simple, universal procedure for evaluating choosing appropriate corrective action doe not exist to deal with situations when one or more QC parameters are outside the acceptance window. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints.

15.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) or Air Force (AFCEE) projects may have additional requirements that are related to USACE laboratory validation (CX), USACE District requirements, government contract requirements, specific USACE project requirements or Architect/Engineer (A/E) requirements. Contact your supervisor for common client requirements (Method Quality Objectives or MQOs, see Appendix 8 for details).

15.1.1 The USACE requirements are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA method may be unattainable for routine sample analysis. Since MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

15.1.2 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality objectives and/or other QC requirements. Exceptions and data qualifications necessary should be documented on the checklists and eventually on case narratives.

15.1.3 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist (see Appendix 7 for details), perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete. For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete. For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director

will review the checklists (initial/date as necessary), prepare case narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

15.1.4 The method blank criteria outlined in USACE MQOs have been modified by USACE auditor(s) as one half of the applicable MQL (personal communications, USACE auditor, 1999).

15.2 HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

15.2.1 WATER - Although EPA 525.2 allows 14-days from sampling for SOCs, the extraction holding time for the laboratory remains at 7-days.

15.2.2 All sample extracts should be analyzed within 23-days after extraction. The total elapsed time since sample collection should not exceed 30-days.

15.2.3 Holding times are established by comparing the dates on the chain of custody for sampling with the dates for extraction (extraction logbook) and extract injection (analysis logbook).

15.2.4 ACTION – If holding times have been exceeded then all positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or invalid (I).

15.3 INSTRUMENT TUNING/PERFORMANCE – Tuning and performance criteria for mass resolution, identification and sensitivity should meet criteria established in ***TABLE 8***. DDT breakdown to DDD and DDE should not exceed 20%. Peak tailing factors for benzidine and pentachlorophenol should be below 3.0 and 5.0 respectively. Perform appropriate corrective action as described in this SOP to restore chromatographic performance to acceptable levels.

15.3.1 Compare mass calibration from tabular report in the raw data with each criteria form.

15.3.2 Check to see if the tuning report is present for each 12-hour period of samples analyzed.

15.3.3 Verify that the spectrum is present.

15.3.4 Calculate % abundance.

15.3.5 ACTIONS – If mass calibration is outside criteria, recalibrate and rerun tuning standard for an acceptable tune report. Reanalyze all associated samples from failed tune. If mass calibration is outside criteria, all associated data may be qualified as estimated (J) or invalid (I) by the client at client's discretion.

15.4 INITIAL CALIBRATION – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the mass spectrometer is capable of producing data within a range that is linear. See also additional client-specific initial calibration requirements including second-source ICV analysis (Appendix 8).

15.4.1 Check to see that the %RSDs for target compounds are within acceptable criteria (% RSD< 30 %) for each individual compound.

15.4.2 ACTION – If more than 10% of the target compounds do not meet the minimum criteria as stated, then correct the underlying problem and re-analyze the ICAL. The calibration is considered invalid.

15.4.3 ACTION – Try linear and/or non-linear regression (requires a minimum of 6-points). The lower limit for correlation coefficient is 0.990 for linear regression.

15.4.4 ACTION – Positive results for compounds not meeting the criteria may need to be qualified as estimated (J) by the user (client). Report detects and non-detects without qualifiers and mention these outliners in the checklist and in the case narrative.

15.5 CALIBRATION VERIFICATION – Calibration verification includes initial calibration verification (ICV) with a second-source standard and continuing calibration verification (CCV) with the same source standard. The ICV is used to check on the accuracy of the primary standard and the CCV is used to check that the instrument is still operating under the linear range of the initial calibration and has remained stable every 12 hours. Check with supervisor for additional client-specific calibration verification requirements (see Appendix 8 for details).

15.5.1 Target Compounds – Check to see that the %D for target compounds is within acceptable criteria – (% D < 30%).

15.5.2 ACTION – If any target compound does not meet the minimum criteria as stated, then correct the underlying problem and re-analyze the CCV. A new ICAL may be ran if necessary.

15.5.3 ACTION - No action is necessary if less than 10% of the target compounds do not criteria and the affected target compound is not detected in the sample.

15.5.4 ACTION – Positive results for samples not meeting the criteria for the CCC and SPCC criteria may be qualified as estimated (J) or invalid (I) by the user (client) at their discretion. Any positive results for compounds other than CCC and SPCC not meeting the criteria may be qualified as estimated (J) by the user (client). Report data without qualifications documenting the QC outliers in the checklist and in the case narrative.

15.6 METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. The processing of blanks helps eliminate reporting of false positives.

15.6.1 Method blanks should be free of all target analytes and other interfering non-target analytes. To be acceptable, blank results should not exceed one half of the MQL for any compounds with the exception of common plasticizers such as phthalate esters.

15.6.2 Review the results of all associated blanks and verify that the method blank has been reported per matrix for each system used to analyze the associated samples. If levels greater than one half the MQLs are present, inspect and correct the problem. Reanalyze all associated samples. The only exception may be quick turn around projects.

15.6.3 ACTION – All positive results are reported with a "B" code if contaminants are found in the method blank.

15.6.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, reanalysis of the samples is not required.

15.6.5 ACTION – Any compound detected in the sample which were also detected in associated method blank both being greater than one half of the MQLs (except phthalate esters) should be re-extracted.

15.6.6 ACTION – If contaminants resulting in GC/MS peak saturation are present, all affected compounds are qualified as invalid (I).

15.7 INTERNAL STANDARD AND SURROGATE RECOVERIES – Sample quality control is performed by means of spiking individual samples with system monitoring compounds which are different structurally, but are chemically similar to classes of target compounds. Four surrogate compounds are spiked into the samples just prior to when the extracting solvent is added to the sample. At least one of the surrogates should be used on routine basis when the target compounds number less than 10. Two or more surrogates should be within the QC limits when the number of target compounds exceeds 20. Since the internal standard is spiked into the sample before extraction, its recovery is calculated using a recovery standard, which is added to the final extract.

15.7.1 Verify internal standard and surrogate recoveries by comparing recovery limits shown in ***TABLE 9*** with the observed recoveries in the samples.

15.7.2 ACTION – When one or more of the recoveries is outside QC limits, the sample must be re-analyzed. Reanalysis may be performed under dilution if original analysis should indicate high levels of target and/or non-target analytes.

15.7.3 ACTION – When any of the four surrogate recoveries are outside the QC limits for controlled matrices such as MB, LCS and LCSD, it may indicate problems with method performance. Problems should be identified and corrected as soon as possible, preferably prior to the analysis of additional samples. QC criteria described above also applies to these samples.

15.8 LABORATORY CONTROL STANDARD (LCS) – A LCS analysis is used to evaluate method competence in the absence of matrix effects. The LCS is also from the same source as the primary standard (USACE requirement). Since a variety of matrices are encountered during sample processing, the LCS may provide a mechanism to check method efficiency for target compounds without the sample matrix as a factor. Contact supervisor for client-specific guidelines for LCS (see Appendix 8 for details).

15.8.1 The QC limits for LCS/LCSD recoveries are shown in Table 10.

15.8.2 Check the results for the LCS recoveries to see if the results are within the acceptable range. Flag compounds with recoveries outside the QC limits with asterisk (*).

15.8.3 ACTION – No action is taken on LCS recovery data alone to qualify all associated samples. A general rule is to have at least 90% of the target analytes within the control limits. The supervisor should be consulted if this is not achievable. If significant deviations are noticed, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements, which may be compound specific. Check with the project manager about project requirements. Professional judgment may be used by the user in evaluating sample data qualification options in conjunction with other QC data for the project.

15.9 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike data are used to evaluate the precision and accuracy of the analytical method on project samples. Contact supervisor for client-specific guidelines for MS (see Appendix 8 for details).

15.9.1 MS/MSD recovery limits are shown in Table 10. The relative percent difference (RPD) between the duplicate analyses (MS and MSD) is also included in this table.

15.9.2 Check the results for the MS/MSD recoveries. Flag QC outliers with an asterisk (*) on the reports.

15.9.3 Perform one manual calculation using raw data and extraction sheet information.

15.9.4 ACTION – No action is taken on MS/MSD recovery data alone to qualify all associated samples. A general rule is to have at least 90% of the target analytes within the control limits. Since matrix interference could lead to lead to serious analytical problems, the supervisor should be consulted when significant outliers are noted. Document QC outliers for %recovery and RPD on the checklist and on client reports (*) and draw attention to the QC outliers on the case narrative. No action may be taken on MS/MSD recovery data alone either to reanalyze samples and/or to qualify associated samples either by the analyst or by the end user. Professional judgment may be used by the user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

15.10 INTERNAL STANDARD PERFORMANCE – Internal Standard (IS) performance criteria (See also Appendix 9) ensures that the GC/MS sensitivity and response are stable during each run.

15.10.1 IS area counts for CCVs should not vary by more than a factor of 2 (-50% or + 100%) from the associated ICAL standard. The internal standard areas for the current CCV should not differ by ±30% (-30% or + 30%) from the previous continuing calibration verification (CCV) standard.

15.10.2 There are no requirements for the IS areas in samples since internal standards are spiked into samples prior to extraction.

15.10.3 The retention time of the internal standard must not vary by more than 30 seconds from the associated daily calibration standard.

15.10.4 ACTION – Document IS areas and RT on Form-8 equivalents (Appendix 9). If an internal standard is outside of QC limits of the associated daily standard, then re-analysis is required.

15.10.5 ACTION – If an IS retention time varies by more than +/- 30 seconds, the total ion profile must be checked for any false positives or negatives. If a large shift has occurred, then professional judgement must be used as to whether reanalyze or not.

15.11 TARGET COMPOUND IDENTIFICATION – Criteria for GC/MS qualitative analysis (Section 12.1) have been provided to minimize the number of erroneous positive or negative identifications of compounds.

15.11.1 For positive identification of the target compound, its RT must be within +/- 0.06 relative time (RRT) units of the standard RRT.

15.11.2 Check to see if the mass spectra of the sample compound (See Appendix 4 for details) and a laboratory generated standard reference spectrum match according to the following criteria listed in this SOP.

15.11.3 For tentatively identified compounds identification and quantitation, see Section 12.2 (see Appendix 4 for details) for guidelines.

15.11.4 ACTION – Professional judgement must be used when applying the criteria for GC/MS analysis of target analytes. The ion pattern and clusters of the extracted ion profile should match fairly well, taking into account factors such as matrix ions and column bleed.

16.0 REFERENCES

16.1 Method 525.2, Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Gas Chromatography/Mass Spectrometry, J.W. Eichelberger, J.W. Munch, and J.A. Shoemaker, ENVIRONMENTAL MONITORING SYSTEMS LABORATORY OFFICE OF RESEARCH AND DEVELOPMENT, U.S. ENVIRONMENTAL PROTECTION AGENCY, CINCINNATI, OHIO 45268, Revision 1.0, March 1994.

16.2 Methods for the Determination of Organic Compounds in Drinking Water, Supplement II, EPA/600/4-88-039, Method 525.1, Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry, Revision 2.2, July 1991.

16.3 Methods for the Determination of Organic Compounds in Drinking Water, EPA/600/4-88-039, Method 525, Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Gas Chromatography/Mass Spectrometry, J.W. Eichelberger, T.D. Behymer, W.L. Budde, Revision 1.0, 2.0, 2.1 (1988).

16.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), December 1996.

16.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.
16.6 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3500B, Organic Extraction and Sample Preparation, December 1996.
16.7 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3535, Solid Phase Extraction, December 1996.
16.8 Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, EPA, 40 CFR Part 136, Method 625: Base Neutral and Acid Compounds, October 1984.
16.9 AML Standard Operating Procedures, O-8270-RD.
16.10 AML Standard Operating Procedures, Q-WMADISD-RA.
16.11 AML Standard Operating Procedures, Q-MDL-RA.
16.12 AML Standard Operating Procedures, Q-CCGM-RA.
16.13 AML Standard Operating Procedures, Q-Water-RA.

TABLE 1
Chromatographic Retention Times (RT) and Quantitation Ions for Semivolatile Organic Compounds (SOCs) Regulated by the Safe Drinking Water Act (SDWA)

ID	Compound Names	MW (amu)	MCL (ug/L)	CAS Number	R.T. (min.)	Monitored Ions (m/z)		IS
						Quan.	Other	
A	**Target Compounds:**							
1	Alachlor	269	2.0	15972-60-8	12:59	160		IS-2
2	Atrazine	215	3.0	1912-24-9	10:49	200		IS-2
3	Benzo(a)pyrene	252	0.2	50-32-8	25:24	252		IS-3
4	Bis(2-ethylhexyl)adipate	370	400	103-23-1	20:11	129		IS-3
5	Bis(2-ethylhexyl)phthalate	390	6.0	117-81-7	22:11	149		IS-3
6	Hexachlorobenzene	282	1.0	118-74-1	10:27	284	142, 249	IS-2
7	Hexachlorocyclopentadiene	270	50	77-47-4	5:16	237	235, 272	IS-1
8	Pentachlorophenol	264	1.0	87-86-5	11:01	266	264, 268	IS-2
9	Simazine	201	4.0	122-34-9	10:41	201		IS-2
B	**Internal Standards:**							
IS-1	Acenaphthene-d_{10}				7:47	164	160, 162	NA
IS-2	Phenanthrene-d_{10}	188			11:37	188	80, 94	NA
IS-3	Chrysene-d_{12}	240			21:33	240	120, 236	NA
C	**Surrogate Standards:**							
SS-1	1,3-Dimethyl-2-nitrobenzene				5:16	134		IS-1
SS-2	Perylene-d_{12}				27:00	264	260, 265	IS-3
SS-3	Triphenyphosphate				20:25	326		IS-3
SS-4	Pyrene-d_{10}	212			16:41	212	210, 213	IS-2
D	**Recovery Standard:**							
RS-1	Terphenyl-d_{14}				25:00	244	122, 212	IS-3

TABLE 2
Chromatographic Retention Times (RT) and Quantitation Ions for Polynuclear Aromatic Hydrocarbons (PAHs)

TARGET COMPOUND	RETENTION TIME (minutes)	INT STD	Primary (m/z)	Secondary (m/z)
Naphthalene	5:00	IS-1	128	127
Acenaphthylene	7:30	IS-1	152	151
Acenaphthene	7:50	IS-1	154	153
Fluorene	8:59	IS-1	166	165
Phenanthrene	11:41	IS-2	178	176
Anthracene	11:50	IS-2	178	176
Fluoranthene	16:10	IS-2	202	101
Pyrene	16:41	IS-2	202	101
Benzo(a)anthracene	21:31	IS-3	228	114
Chrysene	21:39	IS-3	228	114
Benzo(b)fluoranthene	25:33	IS-3	252	126
Benzo(k)fluoranthene	25:45	IS-3	252	126
Benzo(a)pyrene	25:24	IS-3	252	126
Indeneo(1,2,3-cd)pyrene	30:26	IS-3	276	138
Dibenz(a,h)anthracene	30:32	IS-3	278	139
Benzo(g,h,i)perylene	31:16	IS-3	276	138

TABLE 3
Chromatographic Retention Times (RT) and Quantitation Ions for Phthalate Esters

TARGET COMPOUND	RETENTION TIME (minutes)	INT STD	Primary (m/z)	Secondary (m/z)
Phthalate Esters:				
Bis(2-ethylhexyl) phthalate	22:11	IS-3	149	167, 279
Butyl benzyl phthalate	19:39	IS-3	149	91, 206
Diethyl phthalate	8:68	IS-1	149	150, 177
Dimethyl phthalate	7:13	IS-1	163	164, 194
Di-n-octyl phthalate	18:00	IS-3	149	43, 167
Di-n-butyl phthalate	13:49	IS-2	149	104, 150

TABLE 4
Chromatographic Retention Times (RT) and Quantitation Ions for Chlorinated Pesticides and PCBs

TARGET COMPOUND	RETENTION TIME (minutes)	INT STD	Primary (m/z)	Secondary (m/z)
Chlorinated Pesticides:				
Alpha-BHC	10:19	IS-2	181	
Beta-BHC	10:57	IS-2	181	
Gamma-BHC	11:13	IS-2	181	
Delta-BHC	11:57	IS-2	181	
Heptachlor	13:19	IS-2	100	
Aldrin	14:24	IS-2	66	
Heptachlor Epoxide	15:34	IS-2	81	
Gamma-Chlordane	16:19	IS-2	373	375
Endosulfan I	16:44	IS-2	195	
Alpha-Chlordane	16:43	IS-2	373	375
Dieldrin	17:35	IS-3	79	
DDE	17:20	IS-3	246	
Endrin	19:15	IS-3	81	67, 263
Endosulfan II	18:35	IS-3	195	
Endrin Aldehyde	19:02	IS-3	67	250, 345
DDD	18:40	IS-3	235	165
Endosulfan Sulfate	19:47	IS-3	272	
DDT	19:52	IS-3	235	165
Endrin Ketone	19:00	IS-3	317	67, 319
Methoxychlor	21:36	IS-3	227	
Chlordane, Technical	NA	IS-3	373	375
Toxaphene, Technical	NA	IS-3		
Polychlorinated Biphenyls (PCBs):				
Aroclor - 1016	NA	IS-2	152, 256, 292	222, 260
Aroclor - 1221	NA	IS-2	190	224, 260
Aroclor - 1232	NA	IS-2	190	224, 260
Aroclor - 1242	NA	IS-3	222	256, 292
Aroclor - 1248	NA	IS-3	292	362, 326
Aroclor - 1254	NA	IS-3	220, 326, 360	292, 362
Aroclor - 1260	NA	IS-3	326, 360, 394	362

TABLE 5
Chromatographic Retention Times (RT) and Quantitation Ions for Miscellaneous Compounds

TARGET COMPOUND	RETENTION TIME (minutes)	INT STD	Primary (m/z)	Secondary (m/z)
Ametryn				
Atraton				
Bromacil				
Butachlor				
Butylate				
Carboxin				
Chlorneb				
Chlorobenzilate				
Chlorpropham				
Chlorpyrifos				
Chlorthalonil				
Cyanazine				
Cycloate				
Diazinon				
Dichlorvos				
2,4-Dinitrotoluene				
2,6-Dinitrotoluene				
Diphenamide				
Disulfoton				
Disulfatone sulfone				
Disulfoton sulfoxide				
EPTC				
Ethoprop				
Etridiazole				
Fenamiphos				
Fenarimol				
Fluridone				
Hexazinone				
Isophorone				
Merphos				
Metolachlor				
Methyl paraoxon				
Metribuzin				
Mevinphos				
Molinate				
Napropamide				
Norflurazon				
Pebulate				
Permethrin, cis				
Permethrin, trans				
Prometon				
Prometryn				
Pronamide				
Propachlor				
Propazine				

TABLE 6
Preparation of Standards for Initial Calibration (ICAL)
(Table 1 Target Analytes)

ID	Compound Names	Stock ID	Conc. µg/mL	ICAL Concentrations (µg/mL)					
				1	2	3	4	5	6
A	**Target Compounds:**								
1	Alachlor	EPA-1068	5000	0.2	1.0	2.5	5.0	10.0	20.0
2	Atrazine	EPA-1176A	1000	0.2	1.0	2.5	5.0	10.0	20.0
3	Benzo(a)pyrene	SVM-500	500	0.2	1.0	2.5	5.0	10.0	20.0
4	Bis(2-ethylhexyl)adipate	SVM-500	500	0.2	1.0	2.5	5.0	10.0	20.0
5	Bis(2-ethylhexyl)phthalate	SVM-500	500	0.2	1.0	2.5	5.0	10.0	20.0
6	Hexachlorobenzene	SVM-500	500	0.2	1.0	2.5	5.0	10.0	20.0
7	Hexachlorocyclopentadiene	SVM-500	500	0.2	1.0	2.5	5.0	10.0	20.0
8	Pentachlorophenol	SVM-500	500	0.2	1.0	2.5	5.0	10.0	20.0
9	Simazine	PP-530A	100	0.2	1.0	2.5	5.0	10.0	20.0
B&C	**Internal Standards and Surrogates:**								
IS-1	Acenaphthene-d_{10}	ISM-510-1	500	5.0	5.0	5.0	5.0	5.0	5.0
IS-2	Phenanthrene-d_{10}	ISM-510-1	500	5.0	5.0	5.0	5.0	5.0	5.0
IS-3	Chrysene-d_{12}	ISM-510-1	500	5.0	5.0	5.0	5.0	5.0	5.0
SS-1	1,3-Dimethyl-2-nitrobenzene	ISM-510-1	500	5.0	5.0	5.0	5.0	5.0	5.0
SS-2	Perylene-d_{12}	ISM-510-1	500	5.0	5.0	5.0	5.0	5.0	5.0
SS-3	Triphenyphosphate	ISM-510-1	500	5.0	5.0	5.0	5.0	5.0	5.0
SS-4	Pyrene-d_{10}	ISM-510-1	500	5.0	5.0	5.0	5.0	5.0	5.0
D	**Recovery Standard:**								
RS-1	Terphenyl-d_{14}	ATS-160-1	500	5.0	5.0	5.0	5.0	5.0	5.0

TABLE 7
Preparation of Standards for ICV and CCV
(Table 1 Target Analytes)

ID	Compound Names	Source Of ICV STD	Stock ID	Stock Conc. µg/mL	Source of CCV STD	Stock ID	Stock Conc. µg/mL	ICV & CCV Conc.
A	**Target Compounds:**							µg/mL
1	Alachlor	Ultra	EPA-1068	5000	Supelco	41089	1000	5.0
2	Atrazine	Ultra	EPA-1176A	1000	Supelco	48187	1000	5.0
3	Benzo(a)pyrene	Ultra	SVM-500	500	Supelco	47400-U	500	5.0
4	Bis(2-ethylhexyl)adipate	Ultra	SVM-500	500	Supelco	47400-U	500	5.0
5	Bis(2-ethylhexyl)phthalate	Ultra	SVM-500	500	Supelco	47400-U	500	5.0
6	Hexachlorobenzene	Ultra	SVM-500	500	Supelco	47400-U	500	5.0
7	Hexachlorocyclopentadiene	Ultra	SVM-500	500	Supelco	47400-U	500	5.0
8	Pentachlorophenol	Ultra	SVM-500	500	Supelco	48692	500	5.0
9	Simazine	Ultra	PP-530A	100	Supelco	47400-U	500	5.0
B& C	**Internal Standards and Surrogates:**							
IS-1	Acenaphthene-d_{10}	Ultra	ISM-510-1	500	Ultra	ISM-510-1	500	5.0
IS-2	Phenanthrene-d_{10}	Ultra	ISM-510-1	500	Ultra	ISM-510-1	500	5.0
IS-3	Chrysene-d_{12}	Ultra	ISM-510-1	500	Ultra	ISM-510-1	500	5.0
SS-1	1,3-Dimethyl-2-nitrobenzene	Ultra	ISM-510-1	500	Ultra	ISM-510-1	500	5.0
SS-2	Perylene-d_{12}	Ultra	ISM-510-1	500	Ultra	ISM-510-1	500	5.0
SS-3	Triphenyphosphate	Ultra	ISM-510-1	500	Ultra	ISM-510-1	500	5.0
SS-4	Pyrene-d_{10}	Ultra	ISM-510-1	500	Ultra	ISM-510-1	500	5.0
D	**Recovery Standard:**							
RS-1	Terphenyl-d_{14}	Ultra	ATS-160-1	500	Ultra	ATS-160-1	500	5.0

Table 8
DFTPP Specifications for Mass Abundance
(EPA 525.2 and SW-826 Method 8270C)

Mass	Method 525.2 Criteria For Relative Ion Abundance	Method SW-846 8270C Criteria For Relative Ion Abundance
51	10-80% of mass 198	30-60% of mass 198
68	<2% or mass 69	<2% or mass 69
70	<2% or mass 69	<2% or mass 69
127	10-80% of mass 198 or base peak	40-60% of mass 198
197	< 2% of mass 198	< 1% of mass 198
198	Base peak, or >50% of 442	Base peak, 100% relative abundance
199	5-9% of mass 198	5-9 5 mass 198
275	10-60% of mass 198	10-30% of mass 198
365	> 1% of mass 198	> 1% of mass 198
441	Present but less than mass 443	Present but less than mass 443
442	Base peak or >50% of mass 198	> 40% of mass 198
443	15-24% of mass 442	17-23% of mass 442

Table 9
Internal Standard Recoveries
Surrogate Recoveries

Compound	Environmental Samples Limits for Percent Recoveries		MB, LCS/LCSD Samples Limits for Percent Recoveries	
	Lower	Lower	Upper	Upper
Internal Standards: (relative to Recovery Standard)				
Acenaphthene-d_{10}	45	135	70	130
Phenanthrene-d_{10}	45	135	70	130
Chrysene-d_{12}	45	135	70	130
Surrogate Standards: (relative to Internal Standard)				
1,3-Dimethyl-2-nitrobenzene	70	130	70	130
Perylene-d_{12}	70	130	70	130
Triphenyphosphate	70	130	70	130
Pyrene-d_{10}	70	130	70	130

Table 10
Aqueous MDLs, MQLs, and QC Limits for LCS and MS

SDWA Semivolatile Organic Compounds (SOCs) Analysis by GC/MS

Analytical Management Laboratories

Matrix: Water	Preparation Method			525.2											
	Analytical Method			525.2											
	Spike	Mean	S.D.	**MDL**			**MQL**			**LCS Limits**		**MS Limits**			**RPD**
Compound	Amount	ug/L	Ug/L	**ug/L**			**Ug/L**			Lower	Upper	Lower	Upper	Limit	
	Ug/L	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%	
Alachlor	0.5	0.47	0.056	**0.16**			1.0			60	120	45	135	50	
Atrazine	0.5	0.54	0.026	**0.078**			1.0			60	120	45	135	50	
Benzo(a)pyrene	0.5	0.58	0.011	**0.032**			0.2			60	120	45	135	50	
Bis(2-ethylhexyl)adipate	0.5	0.42	0.030	**0.090**			1.0			60	120	45	135	50	
Bis(2-ethylhexyl)phthalate	0.5	0.42	0.030	**0.090**			1.0			60	120	45	135	50	
Hexachlorobenzene	0.5	0.39	0.039	**0.13**			1.0			60	120	45	135	50	
Hexachlorocyclopentadiene	0.5	0.43	0.024	**0.072**			1.0			60	120	45	135	50	
Pentachlorophenol	0.5	0.55	0.092	**0.30**			1.0			60	120	45	135	50	
Simazine	0.5	0.55	0.046	**0.15**			1.0			60	120	45	135	50	

LCS and MS %R limits are in accordance with USACE EM200-1-3, Appendix I.

Sporadic marginal failure (SMF) limits for MS recoveries are 15% to 150%.

Sporadic marginal failure (SMF) limits for LCS recoveries are 15% to 150%.

Sporadic marginal failure (SMF) limit for LCS, MS or sample duplicates is 60%.

Number of SMF compounds allowed: 1

Table 11
List of Appendices

Appendix Number	Appendix Topic
1	Chromatographic Conditions (Instrument Printouts) DFTPP Tune Summary (Form-5 equivalents)
2	Initial Calibration (ICAL) Summary (Form-6 equivalents)
3	Calibration Verification (ICV and CCV) Summary (Form-7 equivalents)
4	Instrument Printouts Quantitation Reports, Sample Chromatograms, and Spectral Identification Library Search for tentatively Identified Compounds (TICs)
5	Aqueous Sample Reports (Form-1 equivalents)
6	Method Detection Limits (MDLs) Study Results
7	Checklist for Data Verification
8	USACE Method Quality Objectives MQOs) for Method 525.2 (USACE, AFCEE, etc. MQOs)
9	Internal Standard Area and RT Reports (Form-8 equivalents)
10	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
11	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
12	Internal Standard and Surrogate Recovery Summaries (Form-2 equivalents)
13	QC Association Forms (Form-4 equivalents) Sample Preparation Log Instrument Analysis Log

NOTE:
Appendices cited in this document contain graphics, hand-written note s, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

INSTRUCTIONS FOR HANDLING AND PROCESSING SAMPLES COLLECTED IN VOA VIALS FOR SW-846 METHOD 5035
(Excerpts from the AML SOP)

Soil samples collected for VOCs and/or GRO analyses using AML SOP Option A to C are contained in three 40-mL VOA vials and one cut-off syringe for each sample. The tare weight of each vial, which includes the cap, magnet, preservative (when applicable), and the sample label is recorded on the label using indelible ink. Weight is recorded to two decimal places (four significant numbers) by AML. The weight of the syringe is not recorded.

Instructions to Field Personnel:

1. Do not attach additional labels to any of the 40-mL vials. The labels will add additional weight (0.3 to 0.7-g depending on the label) to the vial. <u>If you attach any label to the vial, you must re-weigh the vial in the field before and after adding the soil sample and record the weights or the sample weight only on the new label.</u>
2. You may attach a label to the syringe. Attach additional labels containing detailed sample information (project name, sample location, field sample number, date, time, sampler, etc.) to the bubble-wrap pouch that contains the three 40-ML vials and the syringe and seal the pouch with transparent tape.
3. Label each sample vial with the field sample number using indelible ink.
4. Add approximately 4 to 6-g soil to each vial without splashing and/or spilling the preservative. Do <u>NOT</u> leave the vial containing methanol open for extended periods of time to minimize solvent evaporation.
5. The outside of the vials and the caps should be clean. Dirt will add additional weight to the vials, which does not represent sample weight used in the analysis.
6. The threads should be clean and free of dirt. Otherwise, the vials may not seal properly and the volatile organic compounds may escape from the vial.
7. If possible, weigh the vial in the field to two decimal places and record it directly on the vial. Ship the samples as quickly as possible to the laboratory.

Instructions to Laboratory Sample Receiving Personnel:

1. After initial inspection, record laboratory sample number on each vial and the syringe using indelible ink.
2. Allow the sample vials to reach room temperature before weighing each vial to two decimal places (to four significant numbers) and recording them on the vial using indelible ink. Do NOT open any of the vials. The sample custodian may attach additional stick-on labels to the vials after recording the sample weight.
3. AML personnel should complete Form-C if samples are preserved in the field. AML personnel should complete Form-A if samples are frozen (Plan A) within 48-hours of sample collection. AML personnel should complete Form-B if samples are preserved in the laboratory by injecting sodium bisulfate solution (A) or methanol (M) through the septum (Plan B) within 48-hours of sample collection. Other laboratories (e.g. QA Lab) should use their own forms and procedures to collect and record the information.
4. Check and tighten the cap on the vials before storing them in a freezer (Plan A) or refrigerator (Plan B and C).

Detailed Instructions:

See excerpts from the AML SOP on Plan A to C (attached) for additional details.

AML Plan A

This is the preferred option. This option will be used by AML as the default option, when other options are not specified in the FSP or SAP. Balances and preservatives are not required in the field for this option. Under this option, samples are collected in the field and shipped immediately so that they are received by the laboratory within 48-hours of collection. Upon receipt, the samples are examined, weighed, logged in and stored frozen at -12°C until analysis.

1. For each soil or sediment sample to be collected, assemble a kit, which consists of the following: three pre-weighed and labeled 40-ml VOA vials, each containing a 0.125" magnetic stirrer, one EasyDraw Syringe®, and one foam pouch. For each sampling event, assemble a kit, consisting of the following: one Powerstop Handle® syringe holder; cooler(s) of appropriate size; trip blank(s), and zip-lock bag containing chain of custody forms, sample labels, custody seal, etc.
2. The syringe holder will have settings for collecting 5-gram, 10-g or 13-gram samples for different analyses. Remove cap from the syringe. Insert the syringe into one of the three (heavy, medium, and light) 5-g positions. Push the syringe into the representative soil being sampled until the soil column inside the syringe has forced the plunger to the stop point (top of the syringe holder). Remove the syringe from the holder, wipe the exterior of the syringe with a paper towel, insert syringe into one of the open pre-weighed 40-ml vials and eject sample into it. Avoid getting dirt on the threads of the 40-ml vial. Do not remove the stirring bar from the vial. Wipe the vial threads if necessary, cap vial immediately, and store at 4°C.
3. Collect two additional 40-mL vials for each sample using procedures described in Section 8.1.2. Use the same syringe for collecting additional vials.
4. Insert the syringe into the lowest (13-g) position. Collect an additional aliquot for the determination of percent solids (see Section 2.3.1) and cap syringe.
5. Upon receipt at the laboratory, the sample custodian will assign a laboratory sample number for each sample and labels such as A, B, and C to each of the three 40-ml vials and document them in Form-A. The sample custodian will copy into Form-A the tare weight of each vial that has been pre-determined and recorded on the vials. The sample custodian will determine the weight of the vial plus sample to two decimal places and record it on Form-A. The sample weight is the difference between the two weights.
6. Tighten the cap on the vials, if necessary and store them at -12°C in a freezer. Enter "F" for the type of preservation for all the vials in Form A. ***NOTE: The tightening step is necessary, since the rubber seal may shrink when the vials are frozen. Vapor-tight seal must be maintained during storage.***
7. Label the syringe appropriately and store them at 4°C in the walk-in cooler along with other project samples.
8. Immediately before analysis, the analyst will allow one (or more vials at analyst's discretion) of the vials to thaw and warm up to room temperature. The analyst will perform analysis as described in the respective analytical method without the addition of preservatives for low level method. The vials will never be opened in the laboratory.
9. If the results of the initial analysis indicate the need for medium level analysis, sample contained in one of the three vials will be preserved with methanol. Enter date of preservation and type ("M" in addition to "F") for methanol extraction/preservation of the affected sample vials in Form A. Include volume of methanol in Form A. The default volume is 5.0-ml. Volumes (ml) that are equal to the wet sample weight (g) may be used at analyst's discretion. See SOP for the respective analytical method for medium level analysis.

AML Plan B

1. This option (Plan B) is identical in sample collection to Plan A. Balances and preservatives are not required in the field for this option. Under this option, samples are collected in the field and shipped immediately so that they are received by the laboratory within 48-hours of collection. Upon receipt, the samples are examined, weighed, logged in, preserved and stored at 4°C until analysis.
2. See Sections 1 to 4 in Plan A for field sample collection.
3. See Section 5 of Plan A for the determination of sample weights. Record weights in Form-B.
4. Using a clean spatula, transfer approximately 1 g of the sample from the syringe to a labeled, clean 5-mL screw cap vial. Add 1-mL of the $NaHSO_4$ reagent. Seal the vial tight with a screw cap, shake and set aside. Observe for effervescence and record it on the "Comments-lab use only" column in Form-B. After approximately 1 hour, open the vial looking for pressure build-up. Record date and any observations such as "pressure", "effervescence", etc. in Form-B. If neither effervescence nor pressure build-up is noticed, two of the vials for each sample can be preserved with acid for low concentration analysis. **NOTE:** If the sample reacts with $NaHSO_4$, preservation of the affected samples with this reagent should be abandoned. Use Plan A for sample preservation and analysis. Enter "F" for the type of preservation for the affected sample vials in Form B. The sample custodian may seek the help of analyst to perform the acid compatibility tests.
5. $NaHSO_4$ preservation of samples for low level analysis should be performed by injecting 5-mL of the $NaHSO_4$ solution (See laboratory SOP for preparation of this reagent) into two 40-mL VOA vials for each sample using a 5-mL syringe equipped with a sharp 22-gauge needle. Do ***NOT*** use heavier gauge needles or needles that are damaged or defective for preserving the integrity of the septum seal. Record the preservative (A) and its volume in Form-B. Store vials at 4°C until the analyses are complete.
6. Methanol preservation of samples for medium or high level analysis should be performed by injecting 5-mL of the purge and trap grade methanol into the third 40-mL VOA vial using a 5-mL syringe equipped with a sharp 22-gauge needle. Do ***NOT*** use heavier gauge needles or needles that are damaged or defective. Record the preservative (M) and its volume in Form-B. The default volume for methanol is 5.0-mL. The sample custodian may use volumes (ml) that are equal to the wet sample weight (g) if directed by the analyst. The analyst may open the 40-mL vial and transfer the methanol extract to an appropriately labeled 5-mL vial for convenience. Store vials at 4°C until the analyses are complete.
7. Immediately before analysis, the analyst will allow the vials to warm up to room temperature and perform analysis as described in the respective analytical method. The $NaHSO_4$ preserved vials will never be opened in the laboratory.

AML Plan C

Plan C - This option (Plan C) is identical in sample collection to Plan A. Balances and preservatives are required in the field for this option. Under this option, samples are collected into vials containing preservatives. Upon receipt, the samples are examined, weighed, logged in, and stored at 4°C until analysis.

1.0 For each soil or sediment sample to be collected, assemble a kit consisting of the following:
- 1.1 Two pre-weighed (weight=VP) and labeled 40-ml VOA vials, each containing a 0.125" magnetic stirrer and $NaHSO_4$ preservative;
- 1.2 One pre-weighed (weight =VP) and labeled 40-ml VOA vial containing the methanol preservative;
- 1.3 One EasyDraw Syringe®; and
- 1.4 One foam pouch.

2.0 For each sampling event, assemble a kit, consisting of the following:
- 2.1 One Powerstop Handle® syringe holder;
- 2.2 Cooler(s) of appropriate size;
- 2.3 Trip blank(s);
- 2.4 Zip-lock bag containing chain of custody forms, Form-C, sample labels, custody seals, etc.

3.0 The field kit (responsibility of the sampler) should also contain a portable balance and additional glassware to perform field reactivity tests. The laboratory is not responsible for providing these items to non-AML personnel.
- 3.1 Perform acid reactivity tests for each sample in the field prior to adding soil samples into vials containing sodium bisulfate or methanol. (**NOTE:** Have additional vials without preservatives ready for sample preservation, if acid preservation needs to be abandoned.
- 3.2 The laboratory does not provide additional vials to non-AML personnel unless specifically requested by the client. Additional charges will apply.
- 3.3 Weigh the vial (with cap) plus sample (FW) to two decimal places in the field and record the weight on the sample label.
- 3.4 Store sample vials at 4°C until samples are ready for shipment.

4.0 Upon receipt of samples at the laboratory, the sample custodian will assign a laboratory sample number for each sample and labels such as A, B, and C to each of the three 40-ml vials. The sample custodian will wipe the vials dry and determine the weight of each vial plus sample (LW) to two decimal places for quality assurance purposes and record them on Form-C submitted by the field samplers. Store vials at 4°C until the analyses are complete.
- 4.1 If the difference (FW-LW) between the weights recorded in the field and the laboratory is greater than 0.2-g, the integrity of the sample (due to potential leaks during shipment) or field gravimetric measurements may be suspect. Corrective actions, which normally involve re-sampling may be necessary for the affected samples.
- 4.2 If the field weight (FW) is unavailable, leave this column in Form C blank. Contact the client Project Manager and/or the sampler for guidance. The laboratory will adhere to decisions made by the Project Manager. Calculate sample weights (S) using laboratory-determined weight (LW-VP) instead of the field-determined weight (FW-VP).
- 4.3 The $NaHSO_4$ preserved vials will never be opened in the laboratory. The analyst may open the 40-ml vial intended for medium level method and transfer the methanol extract to an appropriately labeled 5-ml vial for convenience.

Analytical Management Laboratories

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FORM-A VOC Soil Sample Collection and Preservation Record per AML Plan A (SW-846 Method 5035)

CLIENT PROJECT & NO: ____ AML WORK ORDER NO: ________________

Date (log-in):						Date of methanol extraction: (when applicable)	
Field Sample Number	Lab. Sample Number	Vial Number	Weight of vial only (g)	Wt. of vial Plus sample (g)	Sample Wt. (grams)	Preservation F = Freezing	Comments (lab use only)
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
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						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	
						F	

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FORM-B VOC Soil Sample Collection and Preservation Record per AML Plan B (SW-846 Method 5035)

CLIENT PROJECT & NO: ____ AML WORK ORDER NO: _______________

Date (log-in and preservation):

Field Sample Number	Lab. Sample Number	Vial Number	Weight of vial only (g)	Wt. of vial Plus sample (g)	Sample Weight (g)	Preservation Method & Volume (ml) A = Acid M = Methanol	Comments (lab use only)

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FORM-C VOC Soil Sample Collection and Preservation Record per AML Plan C (SW-846 Method 5035)

CLIENT PROJECT & NO: ______________________________ AML WORK ORDER NO: ______________

Date (sample collection): **(field use only)**					Date (log-in): **(lab use only)**				
Field Sample Number (field use only)	Vial Number (field use)	Preservative (ml) A = Acid & M = Methanol (lab/field use)	Weight (VP, g) Of Vial (V) + preservative (P) (lab/field use)	Weight (FW, g) of V + P + sample (S) (field use)	Weight (LW, g) of V + P + sample (S) **(lab use only)**	Wt. Diff. (FW-LW) **(may be NA)**	Sample (S) Weight (g) (LW-VP) **(lab use)**	Lab. Sample Number **(lab use only)**	Comments **(lab use only)**

STANDARD OPERATING PROCEDURES

No. O-Explosives-RC

NITROAROMATICS, NITRAMINES, METABOLITES OF EXPLOSIVES AND RELATED COMPOUNDS (Nitoglycerin, PETN, Nitroguanidine) By HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

(EPA SW846-8330A and 8332)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

NITROAROMATICS, NITRAMINES, METABOLITES OF EXPLOSIVES AND RELATED COMPOUNDS (Nitroglycerin, PETN, Nitroguanidine) By HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

(EPA SW846-8330A and 8332)

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) is based on EPA SW-846 Method 8330A, which is used to determine trace levels of explosives compounds in water, soil and sediment by high performance liquid chromatography (HPLC) and ultraviolet (UV) light detector. It is also based on HPLC procedures developed by other organizations such as US Army Corps of Engineers (USACE) and army ammunition plants.

1.2 This SOP is applicable to the standard list of target compounds listed in Table I-A. This method can also be used to metabolites of these compounds (Table I-B) and other compounds listed in Table II-A and Table II-B with appropriate modifications. Samples consisting of various matrices require extraction prior to analysis.

1.3 Detection limits, sensitivity, and optimum ranges of compounds vary with the matrices. All analysts performing this method must have one year of experience in performing HPLC analysis.

2.0 SUMMARY OF METHOD

Samples are extracted prior to analysis by using the methods listed in this SOP. Three sample preparation procedures are applicable for aqueous samples as described in the following sections.

2.1 Low Level Salting-out Method with No Evaporation: Aqueous samples of low concentration are extracted by a salting-out extraction procedure with acetonitrile and sodium chloride. The small volume of acetonitrile that remains undissolved above the salt water is drawn off and transferred to a smaller volumetric flask. It is back-extracted by vigorous stirring with a specific volume of salt water. After equilibration, the phases are allowed to separate and the small volume of acetonitrile residing in the narrow neck of the volumetric flask is removed using a Pasteur pipette. The concentrated extract is diluted 1:1 with reagent grade water. An aliquot is separated on a C-8/Cyano column in tandem, determined at 254-nm, and confirmed on a C-18 reverse phase column.

2.2 Low Level SPE Method: A measured volume (1 liter) of the water sample containing low levels of explosives residues is passed through pre-treated solid phase extraction (SPE) cartridges in accordance with SW-846 Method 3535 or 3535A to trap the explosives in the solid-phase. Target compounds are eluted from the solid-phase using appropriate solvent (methanol or acetonitrile) and analyzed using HPLC as described above.

2.3 High-level Direct Injection Method: Aqueous samples of higher concentration can be diluted 1/1 (v/v) with methanol or acetonitrile, filtered, and analyzed on HPLC as described above. If HMX is an important target compound, methanol is preferred.

2.4 Soil and sediment samples are extracted by placing vials containing homogenized samples and extraction solvent (acetonitrile) in a water-cooled ultrasonic bath with agitation for 18 hours. The bath is kept below room temperature (approx. 60°F) with cold running water. The extracts are filtered and analyzed on HPLC as described above.

2.5 This SOP utilizes a reverse phase HPLC system having an UV absorbance detector system set at 254-nm wavelength for compounds listed in Table I for analyte determination. The wavelength used for compounds listed in Table II-A and II-B are 200-nm and 264-nm respectively. The sample extracts are injected using an auto sampler into the mobile phase stream. The mixture of sample extract and mobile phase are forced with high liquid pressure into a packed column with a C-18 stationary phase or a C-8 column connected in tandem to a Cyano phase, which separate the various compounds according to differences in their partition coefficient and hydrogen bonding. As the individual compound is concentrated and released from the column, it is detected by an absorbance detector, which is electronically attached to a chromatographic output. The compounds are separated according to their column retention time in the form of a chromatographic peak. Either peak area or peak height response can be measured. Peak height is preferred when there is significant peak co-elution.

3.0 INTERFERENCES

3.1 Since the UV detector is a non-specific detector, any organic compound having similar UV characteristics and stationary phase interaction may produce a peak response in the retention time window of the compounds of interest. A second column confirmation must be performed if initial sample extracts produce positive results. Second column confirmation may be waived for specific projects that have been thoroughly characterized in the past. The client, who may take into consideration project needs that may include sample throughput, turnaround time and other requirements, may exercise waiver of the second column confirmation requirement. The analysis is performed using similar instrument conditions, with the analytical column replaced by the confirmation column. It is less likely that the confirmation column will produce the same false positive as the primary column.

3.2 Solvents, reagents, glassware can produce laboratory-generated artifacts, and background interferences, which can produce false positive results. A method blank (MB) analysis is used to verify the absence of these contaminants. If the MB contains any compounds at or above one half of the method quantitation limit (MQL), appropriate corrective action may be necessary.

3.3 Tetryl may decompose with heat or in the presence of methanol / water solution. All aqueous samples suspected to contain tetryl are diluted with acetonitrile prior to filtration. Samples and extracts containing tetryl are stored at 4º C.

3.4 Nitrobenzene and dichloroethane may coelute with nitroglycerine (NG) using the chromatographic conditions described in this SOP. They are likely to be separated on a LC-CN column. Environmental samples monitored at low wavelengths (220-nm or less) for compounds listed in Table II are likely to have additional interferences that are not likely to be problems at higher wavelengths (254-nm) used for compounds listed in Table I.

3.4.1 Glassware used in the extraction procedure is washed in accordance with AML SOP on glassware cleaning (See Section 13, References).

3.4.2 Plastic tubing that comes into contact with aqueous samples during SPE procedure is washed by drawing acetonitrile (HPLC grade) through the tubing for approximately 5-minutes at a flow rate of approximately 5-ml per minute.

3.5 When SPE is used for sample extraction, potentially interfering compounds with weaker affinities for the SPE media can be removed by pre-washing the SPE column with 30% to 40% acetonitrile in reagent water immediately prior to eluting target analytes with 100% acetonitrile.

4.0 APPARATUS AND MATERIALS

4.1 HPLC system

4.1.1 HPLC: Three HPLC systems are used for analysis: Two Agilent 1100 Series High Performance Liquid Chromatography systems, each equipped with data systems, autosamplers, UV/VIS detectors, pumps, controls for purge gases, mobile phase stations, and column ovens; One Hewlett Packard (HP) Model 1090 HPLC, equipped with tunable absorbance detectors (Waters Model 486), Fluorescence detector, A/D converters, circulating cooling bath, and a Chemstation software.

4.1.2 HPLC columns with comparable performance characteristics are available from a number of sources (Phenomenex, Waters, Supelco, etc.). Actual column in use at any given time is subject to change.

4.1.2.1 First (Primary) Column: C-18, 25 cm length, 4.6 mm outer diameter, 5 micron particle size (Phenomenex or Supelco)

4.1.2.2 Second (Confirmation) Column: CN (Supelco or Waters) 25 cm length, 4.6 mm outer diameter, 5 micron particle size, & a C-8 (Waters, Supelco or Phenomenex) 15 cm length, 4.6 mm outer diameter, 5 micron particle size. The C-8 (first dimension) and CN (second dimension) columns are used in tandem for better separation.

4.1.3 The detectors (Waters Model 486) are capable of achieving a 0.001 AUFS sensitivity range at 254-nm and 220-nm wavelengths.

4.1.4 Analog to digital (A/D) converters (Agilent Model 35900E), which are attached to the Agilent Chemstation.

4.1.5 Chemstation for data acquisition and processing (Agilent Rev. A.08.03 (847)).

4.1.6 Ultrasonic bath (Branson 8210) with cold running water to keep bath temperatures below 25º C.

4.1.7 Circulating cooling bath (NESLAB RTE-111) capable of temperature control in the 0°C to 50°C.

4.1.8 HP1090 gradient pump controller used in the isocratic mode.

4.1.9 Labline vortexer (various models).

4.1.10 Analytical balance (Leco LB-8 or equivalent) with a precision of ± 0.0001 g for gravimetric measurements.

4.1.11 Corning stir plate (Various models).

4.1.12 Scientific oven (Various models).

4.1.13 Laser printer(Cannon LBP-860 or equivalent)

4.1.14 Amber scintillation vials (20-ml) or VOA vials (40 ml) with Teflon lined caps for soil sample extraction.

4.1.15 SPE extraction manifolds:

4.1.15.1 (Supelco) with 24-positions for sample extraction, vacuum control and reservoir for spent water sample collection (Kontes Ultraware).

4.1.15.2 (Supelco) with 12-positions for sample extraction, vacuum control and reservoir for spent water sample collection (Kontes Ultraware).

4.1.16 Vacuum pump (Gast Model 2Z866)

4.1.17 Supelco SPE extraction cartridges with C-18 phase; Waters Porapak® R_{DX} C-18 SPE cartridge (Part # WAT047220, reserve lot # W1023B3 or equivalent for one-year supply)

4.1.18 Gelman Science 0.45 um Teflon filter, 47 mm diameter

4.1.19 Hamilton syringes- various sizes

4.1.20 Desiccator

4.1.21 Volumetric flasks-Class A- various sizes

4.1.22 Whatman filtration and degassing unit with 2 liter flask

4.1.23 Air drying box (room temperature oven with a blower fan)

4.1.24 HPLC vials (1.5-ml) from Agilent

4.1.25 Culture tubes, 13 x 100 mm (Fisher)

4.1.26 Eppendorf pipettes, 100-1000 µL and 10-100 µL

4.1.27 Sep-Pak adapter for Waters Porapak® R_{DX} C-18 SPE cartridges

4.1.28 Sep-Pak reservoirs for Waters Porapak® R_{DX} C-18 SPE cartridges

5.0 REAGENTS

5.1 All chemicals used are HPLC grade or better. If contamination is found at or above method detection limit, all suspected chemicals are screened individually and replaced.

5.1.1 Reagent water: Type I water is produced at the laboratory using USFilter water system equipped with a 0.45 micron filter.

5.1.2 Acetonitrile- HPLC grade (EM Science)

5.1.3 Methanol – HPLC grade (EM Science)

5.1.4 Anhydrous Calcium Chloride, reagent grade (Taylor Chemical). Prepare a solution containing 5 g/l in Type I water.

5.1.5 Sodium Chloride- Aldrich Chemicals, reagent grade

5.1.6 Nitroguanidine (NQ) - Aldrich Chemical (Catalogue number N1735-1)

5.2 Stock Standard Solutions (SSS). Stock solutions expire on the dates shown by the vendor, which is generally 1 to 3 years from the date of manufacture. Please note that the expiration date may strictly apply if the container is not opened and stored under manufacturer's recommended conditions (freezer). Do not break the seal if these solutions are not used immediately to prepare other standard solutions. Transfer stock solutions immediately after opening the ampules or vials to Teflon®-lined screw-cap vials and store them in accordance with manufacturer instructions, if any. The stock solutions should be stored in a freezer. Replace standards after 1 year or sooner if signs of degradation (unknown peaks, concentration changes, etc.) are observed. Acquire or prepare only sufficient quantities to last for one year of normal use. Stock solutions are used to prepare intermediate solutions. Several stock solutions are acquired and/or prepared, each with a specific end use: initial calibration (ICAL), initial calibration verification (ICV), continuing calibration verification (CCV), and spiking solutions for laboratory control samples (LCS) and matrix spikes (MS).

5.3 Stock Solution for ICAL, CCV, LCS and MS: A Combined Stock Solution in acetonitrile containing all 14-target compounds listed in Table I at a concentration of 1000-ug/ml is purchased either as a single solution (Ultra Scientific, catalog No. NAIM-833E) or as two solutions (Restek, catalog # 31450 and 31451). Detailed information on the expiry dates of stock standards is included in Appendix 1. Stock solutions expire on the dates shown by the vendor. Store them in a freezer.

5.4 Stock solutions of the individual compounds (See Table in Appendix 1) are purchased from Supelco for definitive compound identification. These are not normally used for preparing calibration solutions for quantitative analysis with the exception of special analytes (PETN, TNG, etc.) as described in Appendix 1. Store them in a freezer. Stock solutions expire on the dates shown by the vendor.

5.5 ICV Stock solution contains target compounds at 10-ug/ml. These are obtained from a second source (Supelco Mix A, cat # 4-7283 and Mix B, cat # 4-7284) at a concentration of 100-ug/ml for each compound. Mix 1-ml of each solution and dilute them to 10-ml with methanol. Store this stock solution immediately in a freezer.

5.6 Surrogate Stock Solution: The compound, 1-Chloro-3-Nitrobenene is used as the surrogate. Stock solution (545-ug/ml) is prepared by weighing 0.0545-g of neat 1-Chloro-3-Nitrobenene (Aldrich, catalog # 218758) and making the solution up to 100-ml with methanol. Store this solution in a freezer. Discard Surrogate Stock Solution 2-years from the date of preparation or sooner if signs of degradation (unknown peaks in the MB, concentration changes, etc.) are observed. ***Note – The analyst may choose a different surrogate (See Table in Appendix 1 for potential surrogate compounds) if the surrogate interferes with target compounds that may be added to this method.***

5.7 Intermediate Standard Solutions (ISS): Intermediate Standard Solutions are prepared from SSS described in Section 5.2. ISS solutions normally expire in 30-days from the date of their preparation. ISS can be kept either in a freezer or a refrigerator. Freezer is preferred if the solutions are not used frequently. If they are used frequently (e.g. almost every day), they should be kept in a refrigerator.

5.8 Surrogate Sample Spike Solution: The surrogate sample spike solution (4000-ng/ml) is prepared by diluting 0.734-ml of the stock solution to 100-ml with methanol. Use 0.5-ml of the Surrogate Sample Spike Solution to spike each aqueous sample (normally 1-L) including all quality control (QC) samples (MB, LCS, MS). Use 1-ml of this solution to spike soil samples (normally 2-g) including all associated QC samples.

5.9 Intermediate ICAL and CCV Standard Solution: This solution, which contains the target compounds and the surrogate at a concentration of 2000-ng/ml is prepared as follows: Dilute 20-ul of the Ultra Scientific (Catalog No NAIM-833E) solution (Section 5.2.1) and 36.7-ul of the Surrogate Stock Solution (Section 5.2.4) to 10-ml of methanol.

5.10 Intermediate ICV Standard Solution: This solution, which contains the target compounds and the surrogate at a concentration of 2000-ng/ml is prepared as follows:

Dilute 1-ml of the ICV Stock Solution (Section 5.2.3) and 18.4-ul of the Surrogate Stock Solution (Section 5.2.4) to 5-ml of methanol.

5.11 LCS/MS Spiking Standard Solution: This solution, which contains the target compounds at a concentration of 4000-ng/ml is prepared as follows: Dilute 100-ul of the ICV Stock Solution (Section 5.2.3) to 25-ml of methanol. Use 0.5-ml of the Surrogate Sample Spike Solution to spike each aqueous sample (normally 1-L) including all quality control (QC) samples (MB, LCS, MS). Use 1-ml of this solution to spike soil samples (normally 2-g) including all associated QC samples. The resulting sample concentrations will be 2.0-ug/L for aqueous samples and 2-mg/kg for soil samples. The final extract concentration will be 200-ng/mL for aqueous (10-mL) and soil sample extracts (20-mL).

5.12 Working Standard Solutions (WSS): All working standards such as initial calibration standards (ICAL), initial calibration verification standards (ICV), and continuing calibration verification standards (CCV) are prepared daily.

5.13 Initial Calibration (ICAL) Standards. Six calibration solutions for Table I-A compounds are prepared using the ICAL/CCV intermediate standard solution (Section 5.3.2) as described below. Use a similar procedure for Table I-B and Table II compounds.

5.13.1 20-STD, which contains 20-ng/ml of each compound, is prepared by diluting 10-ul of the intermediate standard solution (Section 5.3.2) to 1-ml with $CaCl_2$ solution (Section 5.1.4).
5.13.2 60-STD, which contains 60-ng/ml of each compound, is prepared by diluting 30-ul of the intermediate standard solution (Section 5.3.2) to 1-ml with $CaCl_2$ solution (Section 5.1.4).
5.13.3 100-STD, which contains 100- ng/ml of each compound is prepared by diluting 50-ul of the intermediate standard solution (Section 5.3.2) to 1-ml with $CaCl_2$ solution (Section 5.1.4)
5.13.4 200-STD, which contains 200-ng/ml of each compound is prepared by diluting 100-ul of the intermediate standard solution (Section 5.3.2) to 1-ml with $CaCl_2$ solution (Section 5.1.4)
5.13.5 300-STD, which contains 300-ng/ml of each compound is prepared by diluting 150-ul of the intermediate standard solution (Section 5.3.2) to 1-ml with $CaCl_2$ solution (Section 5.1.4)
5.13.6 400-STD, which contains 400-ng/ml of each compound is prepared by diluting 200-ul of the intermediate standard solution (Section 5.3.2) to 1-ml with $CaCl_2$ solution (Section 5.1.4)

5.14 Continuing Calibration Verification Standard (CCV). The CCV standard is identical to the 100-STD described above (Section 5.4.1.3).

5.15 Initial Calibration Verification Standard (ICV). The ICV standard is similar to the 100-STD described above (Section 5.4.1.3) with the exception that it is prepared using a second source intermediate standard solution (Section 5.3.3).

5.16 Mobile Phase: Mobile phase for HPLC analyses are prepared as described below:
5.16.1 HPLC grade water (Type I or II as appropriate)
5.16.2 HPLC grade methanol (MEOH), acetonitrile (ACN) or isopropanol (IP)

5.16.3 For isocratic runs, prepare solvent mixtures at the proportion shown elsewhere (Section 10.1). Degas water, methanol and other mobile phases including mixed solvents by bubbling high purity Helium through the solution for at least 20 minutes.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 One liter amber glass bottles with Teflon lined screw caps may be used for collecting aqueous samples in accordance with EPA SW-846 method 8330. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). See Table III for a summary of requirements. Provide 2 bottles for each water sample to be collected. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. Water samples must be extracted within 7 days of collection.

6.2 One 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples in accordance with EPA SW-846 method 8330. See Table III for a summary of requirements. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each solid sample. Quality control samples such as MS and MSD should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. These samples may be shared with samples collected for other analyses such as semivolatiles (8270C), PCBs (8082), Diesel (DRO, 8015-mod.) and pesticides (8081, 8140, etc.). Air-dried soil samples (for 8330) cannot be used for other tests. Soil and sediment samples must be air-dried and extracted within 14 days of collection.

6.3 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. See Table III for applicable holding times. Analytical data generated outside of the specified holding times must be considered to be minimum values only. The holding time for the extraction of aqueous samples, which includes TCLP leachates is 7 days from the date of sample collection or TCLP leachate preparation. The holding times for the extraction of soil, sediment and waste samples, which includes TCLP leachate preparation is 14 days from the date of sample collection. The holding time for the instrumental analysis of sample extracts is 40 days from the date of extraction. Samples extracts are stored in amber bottles and/or vials at 4 degrees Celsius just prior to analysis. See Table III for more details.

7.0 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control contain a number of regulated compounds at high concentrations. The toxicity of these compounds present in such complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 Proper sample disposal practices (see AML Waste Disposal SOP) should be followed to minimize exposure to toxic compounds.

7.5 Explosives standards and their metabolites are considered dangerous and toxic. Since samples may contain high levels of the explosive compounds, soil samples should not be dried in an oven at temperatures greater than room temperature. Handle samples with caution when preparing and analyzing standards and samples. Use fume hood, gloves and laboratory coats when performing analysis. Samples containing greater than 2 percent TNT must be handled with extra caution, by not grinding these samples and using very small aliquots of the sample. Since lumps of dry chemicals could be raw explosives, contact laboratory manager prior to handling

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Aqueous and soil samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters after the solvent(s) have been allowed to evaporate in a hood.

8.4 Return soil samples containing explosives to the client for proper disposal.

8.5 A

9.0 SAMPLE EXTRACTION PROCEDURE

A sample batch (QC batch, analytical batch, etc. are synonyms) may not exceed twenty (20) environmental samples in addition to the method blank (MB), laboratory control sample (LCS) and a LCS duplicate (LCSD), which is optional. Matrix spike (MS) and MS duplicate (MSD) samples should be counted as environmental samples. Document sample weights, volumes, extraction QC batch number, lot number for the reagents, extract volumes, date, analyst initials, etc. in the sample preparation log book. See Appendix 13 for an example of the sample preparation log. Four aqueous sample extraction procedures are described: the salting-out method (Section 9.1), high level method (Section 9.2), SEP method using Supelco cartridges (Section 9.3.1), and SPE method using Porapak® cartridges. The SPE method using the Porapak® cartridges is the preferred AML procedure for aqueous samples. Several extraction procedures are described. The salting out procedure for aqueous sample extraction is seldom used due to the high solvent consumption and associated waste disposal costs. The solid-phase extraction cartridges are used almost exclusively. A

9.1 Extraction of Low Level Aqueous Samples using the Salting-out Method

9.1.1 Add 250-g of Sodium Chloride to a 1-liter volumetric flask. Measure out 770-ml of sample and transfer to the flask. Add 1-ml of the surrogate sample spike solution. Place flask on stir plate until salt is dissolved. Add 164-ml of acetonitrile (measured with a 250-mL measuring cylinder) while solution is stirring for 15 minutes.

9.1.2 Pipette out the organic layer (approx. 8-mL) into a 100-ml volumetric flask.

9.1.3 Add 10-ml of fresh acetonitrile into extracted sample in 1.0L volumetric and stir for 15 minutes. Repeat procedures 9.1.2 and 9.1.3.

9.1.4 Combine the two organic layers (steps 9.1.2 and 9.1.3).

9.1.5 Add salt water (325-g NaCl /L, approx. 84-mL) to 100-ml mark containing the acetonitrile extract, then stir for approximately 10 minutes and allow the phases to separate (approx. 10 minutes).

9.1.6 Transfer acetonitrile layer to a 10-ml graduated cylinder using a Pasteur pipette.

9.1.7 Add 1-ml of acetonitrile to 100-ml volumetric flask in 9.1.1.2. Stir solution for 10 minutes. Transfer organic layer to the 10-ml graduated cylinder in 9.1.6. Record final volume of the combined final extracts (approx. 5- to 8-mL) to the nearest 0.1 ml mark.

9.2 Extraction of High Level Aqueous Samples. Add 5-ml of sample to a 20-ml scintillation vial. Add 1-ml of the surrogate sample spike solution. Add 4-ml of acetonitrile and shake for 5 minutes. Filter the entire solution through a 0.45-micron Teflon filter disk. Discard the fist 2.5-ml and retain the last 2.5-ml for HPLC analysis.

9.3 Extraction of Low Level Aqueous Samples using the SPE Method. Acceptable recoveries are obtained for two types of cartridges (Section 4.1.15). The Supelco SPE cartridge (Section 9.3.1) used in the past provides acceptable recoveries. The Waters Sep-Pak cartridge (Section 9.3.2), which is currently used by the USACE Environmental Laboratory, Omaha, NE provides improved recoveries for the 14-target compounds as well as non-target explosives metabolite compounds. The Waters cartridges are currently used at AML.

9.3.1 Supelco SPE cartridge: Perform extractions using Supelco SPE vacuum manifold. Condition the SPE tube with 2 to 5-ml of methanol, followed by 2-ml of tap water (NOTE: Do NOT use distilled water or higher grade water since the aqueous conditioner should be similar to the sample in pH, salt concentration, etc.). The tube should not dry up anytime during the conditioning procedure. Disconnect vacuum source before setting up sample extraction. Mark the meniscus on the bottle with a marker. Add 1-ml of the surrogate sample spike solution and mix it thoroughly by gently swirling the bottle. Dip one end of the sample drain tube into the bottom of the sample bottle and snap the other end into the SPE cartridge. Adjust the flow rate of the sample through the SPE cartridge to less than 10-ml/minute by adjusting the vacuum controller for each sample in the manifold. After all the samples have passed through the cartridge, but before the SPE tube goes dry, remove the connecting tube from the cartridge and elute the target compounds with 5.0-ml of methanol. Pulse the vacuum pump to start the flow, then let the methanol drip through under gravity alone at a flow rate of approximately 1-ml/minute. Collect the methanol extract in clean 10-ml screw-cap tubes for instrumental analysis.

9.3.2 Waters Sep-Pak Porapak® R_{DX} C-18 SPE cartridge: Perform extractions using Supelco SPE vacuum manifold. Insert a Sep-Pak adapter (Section 4.1.25) into each Porapak® R_{DX} C-18 SPE cartridge. Connect the cartridges to the vacuum manifold. Connect Sep-Pak reservoirs (Section 4.1.26) to the adapters. Fill each reservoir with 5-ml acetonitrile. Pulse the vacuum pump to start the flow, then let the acetonitrile drip through under gravity alone. Just before each reservoir runs dry, refill the reservoir with 30-ml Type I water. Turn on the vacuum pump and adjust the flow rate to less than 10-ml/minute with the stopcock valves to draw the water through the cartridge. Close valves. The cartridges are now conditioned. Mark the meniscus on the bottle with a marker. Add 1-ml of the surrogate sample spike solution and mix it thoroughly by gently swirling the bottle. Dip one end of the sample drain tube into the bottom of the sample bottle and snap the other end into the SPE

cartridge. Slowly pass the sample through the SPE cartridge by adjusting the vacuum controller for each sample in the manifold to a flow rate of about 10-ml/minute. Empty the vacuum trap as needed. After all the samples have passed through the cartridge, apply full vacuum for a few minutes to remove residual water (NOTE: The SPE tube goes dry in this step). Remove the adapter and reservoir from the cartridge.

9.3.3 Optional cleanup procedure – The SPE cartridge from heavily contaminated aqueous samples may be cleaned up by selectively eluting interfering compounds with weaker affinity for SPE with 30% to 40% acetonitrile in water. Wash the cartridge with 5-mL of 30% (v/v) acetonitrile in reagent water. Discard the washings. **NOTE:** *The nitroaromatics and nitramines may elute from the SPE if the acetonitrile concentration exceeds 40%. Do not use higher volumes of 30% acetonitrile to wash the SPE cartridge.* Set up the vacuum manifold with clean collection vessels (13 x 100 culture tubes, 10-ml capacity) and needles. Elute the target compounds with 5-ml of acetonitrile. Pulse the vacuum pump to start the flow, then let the acetonitrile drip through under gravity alone at a flow rate of approximately 1-ml/minute. Adjust the volume of the extracts to 5-ml with acetonitrile, cap, vortex to mix thoroughly, and store in screw-cap culture tubes for instrumental analysis.

9.4 Soil and Sediment Samples

9.4.1 Thoroughly mix sample as defined in the AML SOP on Subsampling of Containers. Using disposable weigh dishes, weigh out 50-100 grams of wet weight sample and dry in a forced-air oven (Section 4.1.21) at room temperature. After drying, grind and homogenize thoroughly in an acetonitrile-rinsed mortar to pass a 30 mesh sieve.

9.4.2 Weigh out 2.0 ± 0.5 g of dried and homogenized sample into amber 40-ml vials. Spike samples with surrogate solution (1-ml). Spike LCS/LCSD and MS/MSD samples with appropriate spiking solutions (1-ml). Add 10-ml of acetonitrile, cap with Teflon lined cap. Agitate on a vortex mixer for 1 minute.

9.4.3 Place the vial(s) in a water-cooled ultrasonic bath with agitation for 18 hours. Cool the bath with cold running water to keep the bath temperatures below room temperature. It is possible to maintain bath temperatures at approximately 60°F using this procedure.

9.4.4 Allow sample to settle for 30 minutes.

9.4.5 Pipette 10-ml of calcium chloride solution (5 gm/Liter) to the sample vial.

9.4.6 Shake solution for 15 minutes, let stand for a minimum of 15-minutes. Longer times may be needed for samples with fine particles to settle down.

9.4.7 Filter approximately 5-ml through a 0.45 micron Teflon filter disk, discard the first 2 to 3-ml and collect approximately 1- to 1.5-ml in Agilent vials (Section 4.1.22) for HPLC analysis.

9.4.8 A

10.0 INSTRUMENTAL (HPLC) ANALYSIS

10.1 Chromatography Conditions - Follow the instrument manufacturer guidelines and software manuals to set up HPLC to good operating conditions. The C-18 column is used as the primary column for analysis. The C8/CN column is used as the confirmation column. Set chromatographic conditions for the explosives analysis as describe below:

10.1.1 First (Primary) Column Elution Program (C-18) Mobile Phase: Isocratic with methanol:water (55:45) mixtures in both pumps A and B with 0.8-ml/minute flow rate. Mobile phase in both reservoirs is used at equal rates to augment storage capacity for mobile phase. Water is used as the mobile phase for Table II-B compound(s).

10.1.2 Second (Confirmation) Column Elution Program (C-8/CN) Mobile Phase: Isocratic with isopropanol:water (82:18) in both pumps A and B with 1.0-ml/minute flow rate. Mobile phase in both reservoirs are used at equal rates to augment storage capacity for mobile phase.

10.1.3 Columns, mobile phase, temperature, flow rates, etc. may be changed at the analyst's discretion for this and other analyses (e.g. NG/PETN, metabolites, etc.). Run times, flow rates, temperature, injection volume, run conditions, etc. are adjusted at analyst's discretion to optimize chromatographic separation for specific analytes and projects. No changes to the chromatographic conditions should be made between initial calibration and completion of sample analysis. Document all changes and effects since substantial change in peak resolution may require re-calibration of the instrument.

10.1.4 Detector - Waters 486 Tunable absorbance detector set at 254-nm wavelength for compounds in Table I-A and their metabolites (Table I-B). Set the detector at 220-nm for compounds in Table II.

10.2 Calibration

10.2.1 Set up the analysis sequence containing the six calibration solutions for initial calibration (ICAL) on the primary column followed by the analysis of a second source ICV solution. Include calibration blanks as appropriate before and after the analysis of ICAL and ICV solutions. Summarize initial calibration data collected on the primary column as shown in Appendix 2. Using peak areas (or peak heights when appropriate), calculate calibration factors (CF) as described in Section 11.1, the mean calibration factor as described in Section 11.2, standard deviation and percent relative standard deviation (RSD) as described in Section 11.3 for each compound. Use calibration factors from all six ICAL solutions with the exception of 1,3,5-TNB and 1,3-DNB. For these two compounds, the CFs from the highest concentration solution are not used due to non-linearity.

10.2.2 Treat compound pairs that are not separated on the column as a single entity. Calculate calibration factors (identical for both compounds) using the total peak area and the total amount of both compounds.

10.2.3 PROCESSING INITIAL CALIBRATION DATA

10.2.3.1 Linearity – If the % RSD of any compound is 20% or less, then the relative response factor is assumed to be constant over the calibration range and the average relative response factor may be used for quantitation. The analyst, exercising professional judgment, may use regression methods even for compounds with less than 20% RSD, if decreasing or increasing trends are observed for response factors at various concentrations. Since regression methods normally weigh toward high-level standards, weighted regression methods are recommended if the concentration of concern is near low-level standards.

10.2.3.2 If the % RSD of any detected compound is greater then 20%, use least square regression methods to calculate sample concentrations. For regression methods, the instrument response is treated as the dependent variable (y) and the standard amount is the independent variable (x). Do not include origin (0,0) as an extra calibration point.

10.2.3.3 Weighted least squares method is preferred for calculating concentrations more accurately at lower levels. A linear least squares equation ($y = a*x + b$) is preferred over non-linear calibration equations ($y = a*x^2 + b*x + c$). The coefficient of determination (COD or r^2) or correlation coefficient (r) should be used to measure the goodness of fit. For regression models to be used for quantitative purposes, the COD or r^2 must be greater than or equal to 0.99 and r should be $\geq$ 0.995. A value of 1.00 for COD, r^2 and r indicates a perfect fit. Corrective action will be required if the criteria for %RSD, r, r^2, or COD are not met.

10.2.3.4 An initial calibration (ICAL) should be considered a single event process. Consult the QA office or check applicable EPA methods (e.g. SW-846 Method 8000C and 8081A) for additional guidance on appropriate corrective action.

10.2.3.5 Re-fitting of ICAL – The selected model for ICAL may be subjected to an additional re-fitting check to establish representativeness of the model for each compound in each ICAL standard. This is an optional procedure, which is recommended but not required since automated data processing procedures may not be able to accommodate it. Calculate the %D between the calculated amount (Cc) for each compound in each ICAL standard from the ICAL model and its expected value (Ce). Ideally, the %D ((Cc-Ce) * 100 /Ce) for each compound in ICAL standard should meet the QC criteria for %RSD. It is possible for one or more ICAL standards to have %D exceeding the %RSD criteria, while the %RSD for ICAL model is within QC limits. The re-fitting procedure may be used to identify ICAL standards that need re-analysis or dropped from the calculation of ICAL model when re-analysis was not performed.

10.2.3.6 Check and optimize instrument operating conditions including automated peak integration procedures for appropriateness in addressing the chromatographic needs of the specific compound with problems. If the problem appears to be associated with just one of the ICAL standards, that one standard may be reanalyzed once immediately (must be within 8-hours) after the ICAL. Discard all the results from the original analysis of the standard in question and replace it with the results from re-analysis.

10.2.3.7 When additional calibration standards (six or seven instead of five) are analyzed, narrowing the range of concentrations to be used for determining the goodness of fit is an acceptable procedure. Five or more standards should remain for the calculation of RSD or linear regression for each compound after one or more points are removed from the ICAL. More points (6 for second order and 7 for third order) may be needed for non-linear regression. It is generally prohibited to

remove data points from within a calibration range, while still retaining the extreme ends of the calibration range. Do not discard a middle point unless it appears to have an obvious problem and six or more standards were analyzed for ICAL and re-analysis of the affected standard was not performed within the required time limits. Changes to the upper end of the calibration range will affect the need to dilute samples above the range. A change to the lower range is not recommended since it will affect the method quantitation limit (MQL) used to report data. The default MQLs, which are based on the lowest ICAL concentration are stored in the LIMS and project-specific changes are not practical.

10.2.3.8 The refitting procedures may lead to failed %Ds for upper level calibration standards for average response factor methods. The analyst may take alternate corrective actions such as the analysis of sample extracts under dilution for the affected compounds.

10.2.3.9 The refitting procedures may lead to failed %Ds for lower level calibration standards for regression methods. The analyst may take appropriate corrective action such as flagging the sample result as an estimated amount ("J" flag) at the lower level encountered in the sample.

10.2.3.10 **NOTE: Grand mean RSD** – Previous versions of the EPA method(s) had provisions for the calculation of the grand mean %RSD of all target compounds (should be <20% for ICAL to be acceptable) in the ICAL solution, when the % RSD of any of the target compound is greater than 20%. Assessing the validity of ICAL by calculating grand mean RSD is NOT ALLOWED.

10.2.4 The term "calibration verification" (CALV) is used to describe initial verification (ICV) of the initial calibration (ICAL) using second-source standards and subsequent continuing verifications (CCVs) using same-source standards. The response or calibration factors calculated during CALV are not used for sample quantitation since CALV standards are not used as "continuing calibration" standards permitted in some of the EPA methods.

10.2.5 An initial verification (ICV) is performed before any sample extracts are analyzed using the ICAL. For USACE and Air Force (see AFCEE QAPP) projects, a second source ICV (see Section 5.4.3) different from the initial calibration standard should be run immediately after the initial calibration to find any potential systematic errors on calibration standards. Afterwards, continuing calibration verification (CCVs) solutions of the same source is analyzed at the beginning, end, and after every ten samples of an analytical batch. Calculate calibration factor (CF) for each compound as described in Section 11.1 and calculate percent difference (%D) between the CF for ICV and the mean CF from the ICAL as described in Section 11.4. Summarize calibration verification as shown in Appendix 3.

10.2.5.1 The %D for each compound in ICV should be within ±15% (%Rec = 85% to 115%) for each compound according to SW-846, USACE and AFCEE. Refer to Appendix 8 for more information on USACE, DoD QSM, and AFCEE requirements.

10.2.6 Continuing calibration verification (CCV) is performed at the beginning of sample analysis, after every 10 samples and at the end of run using the same source standard (Section 5.4.2) as ICAL. Calculate calibration factors for the daily standard and the %difference (%D) of each from the mean calibration factor from the initial calibration as described in Section 10.2.5 for ICV. Summarize calibration verification collected on the primary and confirmation columns as shown in Appendix 3.

10.2.6.1 The %D for each compound in CCV should be <15%. If the %D for any compound is greater than 15%, the HPLC should be restored to proper operating condition before re-analyzing the CCV for all the compounds. A new ICAL should be generated if the CCV does not meet QC criteria.

10.2.6.2 **NOTE: Grand mean %D** – Previous versions of the EPA method(s) had provisions for the calculation of the grand mean %D for all target compounds (grand mean %D should be <15% for CCV to be acceptable) in the CCV standard, when the %D of any detected compound is greater than 15%. The calculation of the grand mean should be discontinued.

10.2.7 Sample Analysis

10.2.7.1 Set up the following sequence for sample analysis: CCV, MB, LCS, LCSD, MS, MSD, CCV, sample used for MS, 10 samples (including the sample used for MS), and CCV. Repeat CCV and 10 samples and end the run with a CCV.

10.2.7.2 Combine equal volumes (0.5-mL) of the aqueous sample extracts from Section 9.1, 9.2 or 9.3 with calcium chloride solution (0.5-ml, see Section 5.1.4) in a 1.5-ml Agilent HPLC autoinjector vial (section 4.1.22). Filter through a 0.45-micron Teflon filter disk, if necessary.

10.2.7.3 Soil sample extracts prepared as described in Section 9.4.7 contain calcium chloride. These extracts can be injected directly into the HPLC for analysis. Additional dilutions may be necessary if the samples contain high levels of explosives.

10.2.7.4 Analyze samples on the HPLC and calculate sample amounts using mean calibration factors from the initial calibration. Perform calculations as described in Section 11.7 for water samples and Section 11.8 for soil samples. Document analysis on instrument printouts consisting of quantitation reports and chromatograms as shown in Appendices 4 and 5. If none of the target compounds are detected on the primary HPLC column (C-18), samples and associated QC (MB, LCS, MS) may not need to be run on the confirmation column (C8/CN) for rapid turnaround projects (NOTE: Approval should be granted by the Client Project Manager for this option) or routine NPDES monitoring projects.

10.2.7.5 For a compound to be reported as positive, a chromatographic peak must be present within the retention time (RT) window established for the compound. Confirm all positives from the primary column (C-18) on the C8/CN confirmation column (NOTE: see exceptions for special projects, Section 10.3.2). Analyze all associated

QC samples (MB, LCS, MS) in addition to those samples that require confirmation. Calibration and analysis should be performed using procedures described previously for the primary column. Document initial calibration and calibration verification for the confirmation column as shown in Appendices 2 and 3. Document analysis on instrument printouts consisting of quantitation reports and chromatograms as shown in Appendices 4 and 5.

10.2.7.6 Calculate the relative percent difference (RPD) for sample results that are positive between the primary and confirmation columns as described in Section 11.6 for duplicates. The RPD should be 40% or less for data to be reported without qualifiers.

10.2.7.7 The EPA SW-846 Method 8000 (underlying Method for Sw-846 Method 8330) recommends reporting higher of the results obtained from the two chromatographic columns. Although the distinction between the primary and confirmation columns has blurred for most chromatographic (GC and HPLC) analyses, use LC-18 column as the primary column for the explosives analysis. For compounds that are separated from other target compounds on both columns, report the result from the primary column, without a flag if the RPD is <40%. If the RPD is >40% and no visible chromatographic anomalies are present on the primary column, report the primary column result with a "J" flag. If the RPD is >40% and chromatographic anomalies are present on the primary column, report data from the confirmation column with a "J" flag if no chromatographic anomalies are present on the confirmation column. If chromatographic anomalies are present on both columns use professional judgment either to report data from one of the columns or to reject data from both columns (e.g. invalidate result for the compound with an "I" code).

10.2.7.8 For compounds that are separated on one column but not both, report results from the column that separates the target compound from other compounds, with a "J" flag. The professional judgment of the analyst should be used to report data representative of the sample. Document observations in the checklist (Appendix 6) and in the case narrative.

10.2.7.9 If the concentration of any of the target compounds exceed the upper limit of calibration (ICAL), flag the data for affected compounds as estimated ("E" flag). The extracts should be diluted and reanalyzed until the concentrations are within the calibration limits for normal turnaround analyses. For rapid turnaround analyses sample results for compounds that are not contaminants of concern may be reported with "E" qualifiers if sufficient time is not unavailable for reanalysis. Check with Client Project Manager for project specific requirements.

10.2.7.10 The terms, method detection limit (MDL), lowest level for reporting (LLR), reporting limit (RL) and method or practical quantitation limit (MQL or PQL) are defined in the AML SOP on this topic. These are summarized as they apply to the reporting of sample results:

10.2.7.10.1 MDL: Default MDLs for samples prepared and analyzed in strict accordance with default procedures (e.g. soil sample weight = 2.00-g, final extract volume = 20.0-ml, %solids = 100%, and dilution factor =1) are shown in Tables IV, V, VI, VII, and VIII. See Appendix 12 for more details on MDL studies. For actual samples, MDLs are corrected based on the deviation from default values. The corrected MDL value (described with the acronym, SQL) is the lowest amount that can be reported for the sample in question.

10.2.7.10.2 LLR or RL: The lowest level for reporting or reporting limits are defined in the AML SOP on MDLs. These parameters are closely related to the Method Reporting Limit (MRL) that is described in Sections 3.3.7.3 and 3.3.7.4 of the USACE document, EM 200-1-3, Appendix I. For multi-analyte methods, LLRs reported by AML should be uniform round numbers that are equal to or greater than MDLs but less than the MQLs. The MRL defined by USACE can range from MDL to one–half of the applicable project action level, which can be greater than MQL. For risk assessment, USACE recommends setting the LLR at MDL and for other data uses at two times the MDL or higher.

10.2.7.10.3 MQL: Default MQLs for samples prepared and analyzed in strict accordance with default procedures (e.g. water sample volume = 1.00-L, final extract volume = 10.0-ml, and dilution factor =1) are shown in Tables IV to VIII. For actual samples, MQLs are corrected based on the deviation from default values. The corrected MQL value is the lowest amount that can be reported for the sample in question without qualifications as an estimated quantity (J flag). MQLs are normally based on the lowest point of initial calibration (ICAL), which should be at least 3 times the applicable MDL.

11.0 CALCULATIONS

11.1 Calculation of calibration factors (CF):

Tabulate the peak area against concentration for each compound and each standard. Calculate calibration factors (CF) for each compound as shown below:

$$CF = (A_x) / (C_x)$$

where:

A_x = Peak area for the compound being measured

C_x = Concentration of the compound (ng/ml) in the standard

11.2 Calculation of mean calibration factors (mean CF or CF_m):

For each compound, calculate the mean or average calibration factor (mean CF = CF_m) using the five or six CF values calculated for the 5 or 6 standard solutions as shown below:

$$\text{mean CF} = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

Where CF_i are the calibration factors for standards 1 to 5. Calculate the average as shown above using appropriate number of CF_i, if the number of standards (n) analyzed exceeds 5 for specific compounds. Use Excel spreadsheets, laboratory information system (LIMS) or the instrument software to determine mean, standard deviation (See Section 11.3), RSD, linear regression equations, correlation coefficients, etc. Although non-linear equations for the ICAL may be employed in selected instances, its use for routine sample analysis is discouraged.

11.3 Calculation of standard deviations and %RSDs:
Precision for the initial calibration (ICAL) may be expressed as the standard deviation (SD), percent relative standard deviation (RSD), or coefficient of variation (CV). Calculate RSD for the ICAL substituting CF_i for X_i and CF_m for mean x (X_m). The number "n" is the number of standards used to calculate the mean.

$$RSD = CV = \frac{SD * 100}{X_m}$$

$$SD = \left\{\frac{\Sigma(X_i - X_m)^2}{(n-1)}\right\}^{1/2}$$

$$X_m = \frac{\Sigma(X_i)}{n}$$

11.4 Calculation of %D for calibration verification:
Calculate the percent drift or the percent difference (%D) between the observed calibration factor for the verification solution (CF_v) and the mean calibration factor from the ICAL (CF_m) as shown below:

$$\%D = ((CF_v - CF_m) * 100 / (CF_m))$$

Percent drift is calculated when linear regression is used for ICAL. See Section 7.7.1 of Method 8000B for the calculation of %Drift.

11.5 Calculation of Percent Recovery (%R) for LCS/LCSD and, MS/MSD samples:
Percent recovery (%R) is calculated as follows.

$$\%R = \frac{(x_s - x_u)}{K} x\,100$$

where:

X_s = measured sample concentration of the spiked sample

X_u = measured sample concentration of the unspiked sample

K = known concentration of the spike in the spiked sample

When calculating %R for LCS or other reference materials, X_u could be set at zero. The same procedure should be used for calculating surrogate recoveries.

11.6 Calculation of Relative percent difference (RPD) for duplicates:
Precision for duplicate analysis is estimated from the relative percent difference (RPD) of the concentrations (not the recoveries) measured for MS/MSD pairs, LCS/LCSD pairs or for duplicate analyses of unspiked samples. Calculate RPD as shown below:

$$RPD = \frac{|(C_1 - C_2)| * 100}{(C_1 + C_2) * (0.5)}$$

where:
C_1 = Measured concentration of the first aliquot
C_2 = Measured concentration of the second aliquot

Calculate sample concentrations for water and soil samples as described in Sections 11.7 and 11.8 respectively. This equation may also be used to calculate RPD between sample results obtained from dual chromatography columns.

11.7 Calculation of sample concentrations in water samples:
For water samples the calculation is as follows:

$$\text{Concentration } (\mu g/L) = \frac{(Ax * V_e * D)}{(CF_m * V_s)}$$

where:

A_X	=	Peak area of the compound being measured.
CF_m	=	Mean calibration factor ($(ng/ml)^{-1}$) for compound being measured
V_e	=	Volume (ml) of the final extract (default = 10-ml)
V_s	=	Volume of water (ml) extracted (default = 1000-ml)
D	=	Dilution factor for the analysis

11.8 Calculation of sample concentrations in solid (soil, sediment or waste) samples:
For solid samples the calculation is as follows:

$$\text{Concentration } (\mu g/kg) = \frac{(Ax * V_e * D)}{}$$

$$(CF_m * W_s)$$

where:

A_X = Peak area of the compound being measured.
CF_m = Mean calibration factor ($(ng/ml)^{-1}$) for compound being measured
V_e = Volume (ml) of the final extract (default = 20-ml)
W_s = Weight of solid sample (g) extracted (default = 2-g)
D = Dilution factor for the analysis

Solid samples for this analysis are air-dried prior to analysis. Therefore, corrections are not made for %solids in the sample.

12.0 DATA VERIFICATION AND VALIDATION

This section will provide guidance when evaluating and validating data generated using this SOP. Using this SOP will involve implementing the referenced EPA method at the laboratory. Method implementation involves the following: chromatographic separation of the target analytes on two HPLC columns, determination of retention time windows for each compound, determination of method detection limits for each sample matrix and/or preparation method, spiked sample analysis, initial demonstration of acceptable recoveries, etc. Method implementation procedures are described on the AML SOP on MDL. This SOP provides procedures to assess if data produced using this SOP is valid and legally defensible. Document review and comments on one or more checklists or case narratives (See Appendix 6). Verify that sample preparation logs are complete and the instrument run logs are available for review (See Appendix 13 for more details). Anomalies and exceptions should be documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on the checklists. USACE projects may have special requirements (Section 12.1) that will require extra attention. A simple, universal procedure for evaluating and choosing appropriate corrective action doe not exist to deal with situations when one or more QC parameters are outside the acceptance window. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints.

12.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) projects may have additional requirements that are related to USACE laboratory validation (CX), USACE District requirements, government contract requirements, specific USACE project requirements or USACE Architect/Engineer (A/E) requirements. USACE-CX requirements (Summary of Method Quality Objectives for Method 8330) are included in Appendix 8.

12.1.1 The USACE requirements and Department of Defense Quality Systems Manual (DoD QSM) requirements that are shown in Appendix 8 and elsewhere in this SOP are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may or may not

be attainable for routine sample analysis. Since USACE MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

12.1.2 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality objectives and/or other QC requirements. Exceptions and data qualifications necessary should be documented on the checklists and eventually on case narratives.

12.1.3 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist, perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete. For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete. For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director will review the checklists (initial/date as necessary), prepare case narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

12.1.4 The blank criteria shown in Appendix 8 have been modified by USACE as one half of the applicable MQL.

12.2 HOLDING TIMES – The validity of analytical data is based on holding times (See Table III) of the samples from the time of collection to the time of analysis or sample prep.

12.2.1 CHECK- The holding times for extraction of water samples is 7 days from sampling. Soil samples should be extracted within 14-days of collection. TCLP leachates should be prepared within 14-days of soil or waste sample collection. TCLP leachates should be extracted for target compounds within 7-days of leachate preparation. Sample extracts should be analyzed within 40-days of extraction.

12.2.2 CHECK - Actual holding times are determined by comparing the sampling date on the chain of custody with the date of extraction or leachate (TCLP) preparation. Actual holding times for analysis are determined by comparing the extraction date to the injection date. See Table III for applicable holding times.

12.2.3 ACTION – If holding times have been exceeded, contact the client. All positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding

times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or invalid (I).

12.3 INITIAL CALIBRATION – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the instrument is capable of producing data within a range that is linear. See Appendix 2.

12.3.1 CHECK – Check to see if any other compounds are outside the criteria of 20% or less for %RSD. If a compound is outside the 20% criteria, check to see if regression methods are used. The goodness of fit should be measured with the coefficient of determination (COD or r^2), which should be greater than or equal to 0.99. The correlation coefficient (r) should be ≥ 0.995.

12.3.2 ACTION – Check and correct the standards, including the range of concentrations for appropriateness and repeat the ICAL analysis.

12.3.3 ACTION - Report the problem to the Project Manager and report detects and non-detects with appropriate qualifiers for rapid TAT projects. Mention the ICAL anomaly in the case narrative.

12.4 CALIBRATION VERIFICATION – It consists of two parts: initial calibration verification (ICV) described in Sections 10.2.4 and 10.2.5; and continuing calibration verification, which is described in Sections 10.2.6 and 10.2.7. ICV is run immediately after the initial calibration to find any potential systematic errors on calibration standards. CCVs are used to check that the instrument is still operating under the linear range of the initial calibration and it has remained stable by checking before, during, and after sample analysis. See Appendix 3 for documentation of ICV and CCV results.

12.4.1 ICV: Target Compounds - Check to see if %D for any of the target compounds is outside the USACE QC criteria (< ±15%) for ICV, which is also the method criteria for CCV (< ±15%).

12.4.1.1 ACTION - Check the ICAL and ICV standards for integrity. Replace one or more of the working standards (ICV first and ICAL later) for trouble shooting. Correct the problem(s) and repeat ICV analysis. Use professional judgment for trouble shooting for USACE and non-USACE projects.

12.4.1.2 ACTION - When ICAL is acceptable and the ICV fails marginally for a small subset of compounds, positive results and non-detects for target compounds not meeting the ±15% ICV criteria for %D may be reported without data qualifications for non-USACE projects provided a valid CCV was obtained immediately following the ICV.

12.4.1.3 ACTION - When ICAL is acceptable and the ICV fails for any compound, the ICAL and/or ICV standards must be inspected and replaced, if necessary for compliance with this requirement for USACE projects. Perform reanalysis of all affected samples for USACE and other DoD projects. It is good laboratory practice to follow this procedure for all projects.

12.4.2 CCVs: The continuing calibration verifications are used to check that the instrument is still operating under the linear range of the initial calibration and

has remained stable every 12 hours. The first CCV that is run prior to sample analysis should meet the requirements of the ICV without any exception.

12.4.2.1 The %D for compounds present in the sample – The %D for the CCVs before and after the sample analysis should be within QC limits when internal standards are not used. The %D for the CCV before sample analysis should be within QC limits when internal standards are used.

12.4.2.2 The %D for compounds not present in the sample – The %D for the CCVs before and after the sample analysis may be >+15%. Corrective action is not required unless the %D is < -15%.

12.4.2.3 ACTION – Corrective action must be performed for positive results with %D outliers. In exceptional cases (e.g. rapid TAT analysis for screening level data), positive results for compounds not meeting the criteria may be qualified as estimated (J) by the user (client).

12.4.2.4 ACTION - Report non-detects without qualifiers and include a description of anomaly, if any in the case narrative.

12.4.2.5 ACTION – Perform more frequent CCVs (e.g. every 10 samples) than required (every 12-hours) to avoid extensive re-analysis.

12.5 METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Sporadic cases of contamination may be difficult to control. However, daily contamination would not be acceptable. The concentration of target analytes should not exceed default QC criteria for blank (<0.5*MQL) and it should not exceed project-specific QC criteria (<0.5*MRL). If they do, steps shall be taken to find/reduce/eliminate the source of this contamination in the method blank with the exception of common laboratory contaminants (None for this analysis). Additional project-specific corrective actions may be necessary. Sample data qualification to alert the presence of target analytes in the method blank is performed by the laboratory. Sample data qualification based on all blank results (laboratory MB, equipment blank, and other field blanks) is performed by the user (client). The processing of blanks helps eliminate reporting of false positives in project reports. The default laboratory blank criteria (<0.5*MQL) may be identical to project-specific criteria (<0.5*MRL) for most projects. See Tables IV to VIII for a list of MQLs. Since MRLs can range from MDL to one half of the project specific action level (Section 3.3.7.4, Appendix I of USACE document EM 200-1-3), which can be greater than MQL, professional judgment by the analyst will be necessary to apply appropriate QC criteria for the method blank.

12.5.1 CHECK - Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples.

12.5.2 ACTION – Report all positive sample results with a “B” code, if contaminants are found in the method blank at levels greater than the applicable blank criteria for the project.

12.5.3 ACTION – If a compound is detected only in the blank and not in any of the associated samples, then no action is taken. Discuss the situation in the case narrative.

12.5.4 ACTION – Any compound detected in the sample which is also detected in associated method blank at levels greater than the blank criteria will require corrective action. Re-extraction of the associated samples will be necessary unless other client criteria are applicable. Exceptions may include quick turn around projects. Contact client.

12.5.5 ACTION – If the sample amount is more than 10-times the blank amount, re-extraction and re-analysis of the affected sample may not be necessary if the contamination of the MB is associated with grossly contaminated samples.

12.5.6 ACTION - If the sample amounts are less than 10-times the blank level, reanalyze the method blank, and any samples containing the same contaminant. If the contamination remains, the contaminated samples of the batch should be re-prepared and reanalyzed with a new method blank and batch specific QC samples.

12.6 SURROGATE RECOVERIES – Surrogates or system monitoring compounds are similar chemically and in performance to the target compounds in the analytical procedure. These surrogate compounds are added just prior to extraction of the samples and the percent recoveries of the surrogates are calculated and evaluated. A surrogate is evaluated by comparing its recovery (%R) in each sample to the laboratory QC limits, project-specific DQOs, client specifications, or other criteria (e.g. EPA limits). Document surrogate recoveries on Form-2 equivalents (See Appendix 11 for details).

12.6.1 Surrogate failures in method blanks or laboratory control samples are indicative of a general method failure and should be thoroughly investigated. Samples that appear to be heavily contaminated either with target or non-target analytes may require method modification (such as the medium level method instead of low level method) even for an initial analysis. Such requests may either originate from the analyst or the client project chemist. Other consequences such as higher detection limits for the medium level analysis, additional cost for low-level re-analysis, if medium level analysis is unacceptable, etc. should also be considered in this instance. Surrogate recoveries that fail to meet appropriate acceptance criteria would indicate that a potential matrix effect is present. If significant non-target interference occurs, corrective action shall include implementing additional cleanup procedures, and reanalyses. If this does not reduce the interference, discuss the impact on the data within the case narrative. For USACE projects, tighter limits may apply to interference-free samples such as MB, LCS and LCSD.

12.6.2 CHECK - Verify that the surrogate (1-chloro-3-nitrobenzene) recoveries are within QC limits for the environmental samples including MS and MSD (50% to 150%) and interference-free samples including MB, LCS and LCSD (60% to 140% for water and 50% to 150% for soil). Flag outliers with an asterisk (*).

12.6.3 ACTION – When surrogate recoveries are outside control limits, the sample must be re-analyzed. Reanalysis may be performed under dilution if original analysis indicates high levels of target and/or non-target analytes. The SW-846 document does not contain any recommendation for cleanup procedures for Method 8330 analytes. If surrogate recoveries are still outside the limits

after dilution, report both sets of data and describe the problem in the case narrative.

12.6.4 ACTION - If method modifications are needed, contact the client for authorization. The authorization may involve request for additional payment before reanalysis may begin.

12.7 LABORATORY CONTROL SAMPLE (LCS) – The analysis of LCS samples is used to evaluate method competence in the absence of matrix effects. The LCS is evaluated for Method 8330 analytes by comparing the percent recovery for the entire target analytes to the laboratory QC limits, which are identical to the 3-sigma DoD QSM QC limits. See Appendix 10 for guidance on documenting LCS and LCSD concentrations and %R in QC summary report forms. The DoD QSM specifies the number of compounds that can exceed the 3-sigma QC limits based on statistical considerations. The LCS recovery for such compounds should not exceed the marginal exceedance 4-sigma (ME) limits. Evaluation procedures may include comparison of LCS recoveries with those determined by project-specific DQOs (See project QAPP, if available), laboratory statistical control limits, or USEPA guidance (for the non-standard method 8330 method analytes and nitroglycerin). Initially, the effect of QC failure on the samples should be evaluated. Regardless of this assessment, steps shall be taken to find the source of the problem and correct it.

12.7.1 CHECK - LCS and LCSD recoveries (See Appendix 8 for details) should be within QC limits (See Tables IV to VIII). Compare the %R results for each compound in LCS with laboratory QC limits to see if all the results are within the acceptable range. Flag outliers with "ME" if it is outside the 3-sigma control limit but within the 4-sigma control limits. Flag it with "ME*" if it is outside the 4-sigma ME limits.

12.7.2 CHECK - Compare observed %R results for each compound with the laboratory limits for LCS/LCSD recoveries (e.g. USACE criteria). Perform manual calculation for %R, if necessary for QC outliers. Document the number of compounds that exceeded the QC limit and compare it against the maximum recommended by DoD QSM. Document this review in checklists (Appendix 7) and in case narratives.

12.7.3 ACTION - Typically, the LCS would be reanalyzed for the failed analytes (ME*) only. If the second analysis fails, then the LCS, method blank, and all associated samples of the batch would be reanalyzed for the failed analytes only. If sufficient sample is not available for reanalysis, or if the corrective action is ineffective, discuss the situation with the client project manager (or chemist). Include a discussion of the situation in the case narrative. The case narrative should discuss the corrective action taken and any other information.

12.7.4 ACTION - For non-USACE and non-DoD projects, it may be sufficient to provide definitive data of known quality, which includes LCS recovery information with appropriate qualifiers. If significant deviations (e.g. number of target compound exceeding QC limits for recoveries is greater than the allowable maximum) are noticed, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements. Check with the project manager about such requirements. Professional judgment may be used by the user (client) in evaluating and qualifying sample data in conjunction with other QC data for the project.

12.8 MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERIES – Matrix spike recovery data are used to evaluate the accuracy of the analytical method on project samples. The evaluation is performed by comparing the %R for target analytes to the DoD QSM limits or USACE limits established and/or adopted for the LCS samples. Document MS results in Form-1 equivalents and recoveries in Form-3 equivalents (See Appendix 9). Document MS/MSD recoveries in instrument printouts and in LIMS generated reports as shown in Appendix 7. Flag QC outliers with "ME" and "ME*" on client reports. The MS/MSD percent recoveries should be within QC limits (Tables IV to VIII).

12.8.1 Perform one manual calculation using raw data and extraction sheet information. MS data evaluation is more complex than method blank or LCS data evaluation since it is complicated by matrix effects in addition to sample preparation and analysis errors. The heterogeneity of grab soil samples, and potential non-representativeness of sequentially collected water samples, etc. further complicate the evaluation since it is assumed that the native concentrations are constant in triplicate analyses. In addition, concentrations of the target analytes in the sample can also far exceed spike amounts. If the native concentration of target analytes in the sample chosen for spiking is high relative to the spiking concentration, the differences in the native concentration between the unspiked sample and the spiked samples may also become very significant.

12.8.2 In addition to their reluctance to pay for MS and/or MSD analyses, most clients may not know what samples to collect for MS analysis, may not collect sufficient samples, may not have any knowledge of the native analyte concentrations in the chosen sample, and may not have a knowledge of MS spiking levels that are appropriate either for the sample or their project. For procedures with multiple target analytes, spiking levels may be inappropriate for one or more analytes, if samples that contain target analytes are chosen either in the field or by the laboratory. If no project samples are collected for MS analysis, samples from another project used for MS analysis may not be representative of the project samples. When project samples (e.g. surface soil) are chosen, their MS recovery characteristics may be different from those of other samples (e.g. sub-surface soil) in the same batch.

12.8.3 Laboratory project managers should discuss MS requirements with client personnel to ensure that their requirements match with those of their clients and they are achievable on a routine basis by commercial laboratories. Three separate aliquots of samples collected for unspiked and spiked (MS and MSD) sample analyses may be combined, carefully homogenized and subsamples used for the triplicate analysis.

12.8.4 ACTION – The results of the MS are evaluated, in conjunction with other QC information, to determine the effect of the matrix on the bias of the analysis. Steps should be taken to find the cause of failure and corrective action to remedy it. MSs that fail to meet the appropriate acceptance criteria may indicate that a potential matrix effect is present. A review of the MSD result, if available, may confirm the matrix effect, if it is in the same direction and same order of magnitude.

12.8.5 ACTION - If the sample chosen for MS/MSD is inappropriate, notify the client. When additional payments are authorized for re-analysis, reextract and/or reanalyze the MS/MSD samples after employing cleanup procedures (if applicable), dilution techniques to minimize matrix interference, or using other project samples that appear to be clean, when sufficient quantities are available. Re-spike the sample at a higher level (e.g., at approximately two to four times the sample concentration), then reanalyze the sample based on project-specific requirements. It is the responsibility of the client at all times to select appropriate samples for MS analysis, make additional payments for additional analyses, and to offer necessary guidance as needed to meet their data quality objectives.

12.8.6 If the matrix effect cannot be resolved, discuss the situation in the case narrative. No action is taken by the laboratory on MS/MSD recoveries data alone to qualify associated samples. The user (client) may use Professional judgment in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

12.9 DUPLICATES - Duplicates include sample or matrix duplicates (MD), matrix spike duplicates (MS/MSD), and LCS duplicates. The precision is evaluated by comparing the observed RPD between the two sets of results (concentrations and not %R) with laboratory QC limits (50% for soil and water) to see if all the results are within the acceptable range. See Appendices 9 and 10 for guidance on documenting RPDs for LCS/LCSD, MS/MSD and M/MD pairs in QC summary report forms. Flag outliers with an asterisk (*). For USACE projects, acceptance criteria may allow for sporadic marginal failure (SMF) criteria (60%) for a few target analytes (0,1, and 2 respectively) when multiple analytes (<5, 5-15, and 16-30 respectively) are reported for a project. The SMF criteria may also be applied to problem analytes such as tetryl, which has a tendency to decompose during analysis. These criteria should only be applied to concentrations of target analytes that are above each analyte's MQL. MDs or MSDs that fail to meet acceptance criteria may indicate either sample heterogeneity or the presence of potential matrix effects.

12.9.1 CHECK - Compare the RPD results for each compound in the duplicate set (LCS/LCSD, MS/MSD or M/MD) with laboratory QC limits (50% for soil and water and 60% for SMF analytes) to see if all the results are within the acceptable range. Flag outliers with an asterisk (*).

12.9.2 ACTION - LCS duplicates are performed by the laboratory to monitor precision on a routine basis. Reextractions and/or reanalyses are not required if RPDs for LCS/LCSD sets are outside QC limits.

12.9.3 ACTION - The RPDs for MS/MSD and M/MD sets have problems that are similar to that described for MS/MSD %R data. See Section 12.7 for additional guidance. Authorization by client (to cover costs for reanalysis) may be necessary.

12.9.4 ACTION - Check with the project manager about project specific requirements. Professional judgment may be used by the user (client) in evaluating sample data qualification options in conjunction with other QC data for the project.

12.10 TARGET COMPOUND IDENTIFICATION AND REPORTING – In the absence of project-specific criteria, minimize the possibility of incorrect identification (or false positives) through confirmation on a second dissimilar chromatographic column for single component target analytes. After the target analyte has been identified, compare the primary and confirmatory results for agreement according to method-prescribed criteria. Analytical results would normally be reported from the primary column unless interferences were noted. Report sample data using Form-I (See Appendix 14).

12.10.1 CHECK - Positive compounds must be within +/- 0.06 relative retention time (RRT) units of the standard RRT on both columns. The results should be >MDL or other threshold for reporting positives (e.g. LLR, RL) on both columns.

12.10.2 The RPD for sample amounts from the two columns for identified compounds should be within <40% for data to be reported without qualifiers.

12.10.3 ACTION - If the RPD for positive results are <40%, report the result from the primary column without any flag.

12.10.4 ACTION - If the RPD is >40%, report the result from the primary column with a "J" flag. The result from the confirmation column may be reported with a "J" flag at the discretion of the analyst, if interference is suspected for the compound of interest in the primary column.

12.10.5 ACTION – Report target compounds that are present at levels greater than either the MDL or the RL (or LLR as appropriate for the project) using "J" qualifiers if necessary, if the levels are less than MQL. See Section 10.3.10 and the AML SOP for a discussion of MDL, LLR, RL, and MQL.

12.10.6 ACTION - Compounds that were also detected in the associated MB should also be qualified with a "B" code.

12.10.7 ACTION - If the concentration of any of the target compounds exceeds the upper limit of calibration (ICAL), flag the data for affected compounds with an estimated or "E" flag. The extracts may need to be diluted and reanalyzed until the concentrations are within the calibration limits for normal turnaround analyses. For rapid turnaround analyses and for compounds that are not contaminants of concern for specific projects, sample results may be reported with "E" qualifiers. Use additional QC flags as described in the following section.

12.10.8 Data qualifiers shall be added by the analyst and/or the laboratory during data generation, verification, review, or reporting. The following data qualifiers are suggested for normal use:

U - Non-detect when analyte concentration is below MDL or MRL as appropriate

J - Estimated concentration when analyte concentration falls below MQL

J - Estimated concentration when the RPD between the analyte concentrations from the primary and confirmation columns is greater than 40%

B - Blank contamination when any associated blanks are above the MDL or MRL

E- Estimated concentration when analyte concentrations exceed the upper limit of initial calibration

D - Data reported are from method of standard addition (not applicable to this procedure).

I - Invalid results when target and/or non-target interference does not allow a valid result to be reported for a target analyte

R - Rejected due to problems that cannot be resolved by the laboratory within schedule to report meaningful data for the analyte. This flag can only be used by the Level 3 Reviewer.

12.10.9 The QC flags described above may be used to identify any suspected bias in the data, either low or high, and whether the estimation is related to the suspected identification (qualitative) or whether the value reported is an approximation (quantitative). The project manager or appropriate technical personnel should be notified as soon as possible to discuss possible corrective actions for data requiring qualifications. Additional data flagging may be performed during external data review or validation by the client based on project specific requirements, EPA guidance, etc.

13.0 REFERENCES

13.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8330, Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC), September 1994.

13.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8330A, Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC), Revision 1, January 1998.

13.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8332, Nitroglycerine by High Performance Liquid Chromatography (HPLC), Revision 0, December 1996.

13.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.

13.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

13.6 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3500B, Organic Extraction and Sample Preparation, December 1996.

13.7 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3500C, Organic Extraction and Sample Preparation, Draft Update IVB.

13.8 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3535, Solid Phase Extraction, December 1996.

13.9 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3535A, Solid-Phase Extraction, Draft Update IVA and IVB, Draft Revision 1A, November 1998.

13.10 Reversed-Phase HPLC Method for the Determination of NG and PETN in Soil, USACE MRD Laboratory SOP, August 1989.

13.11 EM 200-1-1, Validation of Analytical Chemistry Laboratories, US Army Corps of Engineers. July 1, 1994.

13.12 EM 200-1-2, Technical Project Planning (TPP) Process, US Army Corps of Engineers, August 31, 1998.

13.13 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, Appendix I, US Army Corps of Engineers, 1 February 2001.

13.14 EM 200-1-6, Chemical Quality Assurance, US Army Corps of Engineers, October 10, 1997.

13.15 ER 1110-1-263, Chemical Data Quality Management for Hazardous, Toxic, Radioactive Waste Remedial Activities, US Army Corps of Engineers, April 30, 1998.

13.16 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

13.17 AML Standard Operating Procedures, Q-Water-RC.

13.18 AML Standard Operating Procedures, Q-WasteMgmt-RD.

13.19 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

13.20 AML Standard Operating Procedures, Q-Controlcharts-RC.

TABLE I-A

Standard List of target compounds for SW-846 Method 8330

COMPOUND	Abbreviation	CAS[a] Number
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX	2691-41-0
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX	121-82-4
1,3,5-Trinitrobenzene	135-TNB	99-35-4
1,3-Dinitrobenzene	13-DNB	99-65-0
Methyl-2,4,6-trinitrophenylnitramine	Tetryl	479-45-8
Nitrobenzene	NB	98-95-3
2,4,6-Trinitrotoluene	246-TNT	118-96-7
4-Amino-2,6-dinitrotoluene	4A-DNT	1946-51-0
2-Amino-4,6-dinitrotoluene	2A-DNT	355-72-78-2
2,4-Dinitotoluene	24-DNT	121-14-2
2,6-Dinitotoluene	26-DNT	606-20-2
2-Nitotoluene	2-NT	88-72-2
3-Nitotoluene	3-NT	99-08-1
4-Nitotoluene	4-NT	99-99-0

a Chemical Abstracts Service Registry number

TABLE I-B

Metabolites of target compounds for SW-846 Method 8330

COMPOUND	Abbreviation	CAS[a] Number
2-Methyl-3-nitroaniline or 2-Amino-6-nitrotoluene	2A-6NT	603-83-8
4-Methyl-3-nitroaniline or 4-Amino-2-nitrotoluene	4A-2NT	119-32-4
5-Nitro-ortho-toluidine or 2-Methyl-5-nitroaniline or 2-Amino-4-nitrotoluene	2A-4NT	99-55-8
2,4-Diamino-6-nitrotoluene	4,6-DA-2NT	6629-29-4
2,6-Diamino-4-nitrotoluene	2,6-DA-4NT	59229-75-3

a Chemical Abstracts Service Registry number

TABLE II-A

Additional Explosives Compounds for SW-846 Method 8330

COMPOUND	Abbreviation	CAS[a] Number
Nitroglycerine	NG	55-63-0
Pentaerythritol tetranitrate	PETN	78-11-5
Nitroguanidine	NQ	556-88-7

a Chemical Abstracts Service Registry number

TABLE II-B

Additional Explosives Compounds for SW-846 Method 8330

COMPOUND	Abbreviation	CAS[a] Number
Nitroguanidine	NQ	556-88-7

a Chemical Abstracts Service Registry number

Table III

SAMPLE HOLDING TIMES, DIGESTED VOLUMES AND COLLECTION VOLUMES FOR EXPLOSIVES DETERMINATIONS IN AQUEOUS AND SOLID SAMPLES

Measurement	Final Volume (mL)	Collection Volume (mL or g)	Holding Time Preservation
Water (low, 8330)	10-mL (variable)	1000-mL	7 days prior to extraction, 4º C 40 days from preparation to analysis
Water (low, 3535)	10-mL	1000-mL	7 days prior to extraction, 4º C 40 days from preparation to analysis
Water (High, 8330)	10-mL	100-mL	7 days prior to analysis, 4º C
Waste (TCLP)	2000-mL	500-g	14 days prior to leaching, 4º C
Leachate (low, 8330)	10-mL (variable)	500-mL (variable)	7 days prior to extraction, 4º C 40 days from preparation to analysis
Solid (Total)	20-mL	200-g	14 days prior to extraction, 4º C 40 days from preparation to analysis

Table IV
Aqueous Sample MDL, LLR, MQL and QC Limits for LCS and MS
Standard 8330 List of Compounds

Nitroaromatic and Nitramine Explosives Analysis by HPLC

Analytical Management Laboratories

Preparation Method:	3535A						HPLC Analysis Primary Column					C-18	
Analysis Method:	8330A		Matrix:		Water		Confirmation Tandem Column:					C8/CN	
	MDL	**MDL**	**MDL**	**MDL**	**LLR**	**MQL**	**Ratio (RQ)**		**DoD QSM LCS & MS Control Limits**		**DoD QSM LCS & MS ME Limits**		**RPD**
Compound	Spike	C-18	C8/CN	Report	Report	Report	QL/DL	Flag	Lower	Upper	Lower	Upper	Limit
	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L			**%R**	**%R**	**%R**	**%R**	%
HMX	0.08	0.0204	0.0536	**0.054**	0.1	**0.3**	5.6		80	115	75	120	50
RDX	0.08	0.0127	0.0663	**0.066**	0.1	**0.3**	4.5		50	160	35	180	50
1,3,5-Trinitrobenzene	0.08	0.0090	0.0239	**0.024**	0.1	**0.3**	12.6		65	140	50	150	50
1,3-Dinitrobenzene	0.08	0.0094	0.0104	**0.010**	0.1	**0.3**	28.8		45	160	30	175	50
Tetryl	0.08	0.0224	0.0131	**0.022**	0.1	**0.3**	13.4		20	175	10	200	50
Nitrobenzene	0.08	0.0258	0.0094	**0.026**	0.1	**0.3**	11.6		50	140	35	155	50
2,4,6-Trinitrotoluene	0.08	0.0433	0.0196	**0.043**	0.1	**0.3**	6.9		50	145	35	160	50
4-Amino-2,6-dinitrotoluene	0.08	0.0629	0.0206	**0.063**	0.1	**0.3**	4.8		55	155	40	170	50
2-Amino-4,6-dinitrotoluene	0.08	0.0345	0.0271	**0.034**	0.1	**0.3**	8.7		50	155	35	170	50
2,6-Dinitrotoluene	0.08	0.0285	0.0321	**0.032**	0.1	**0.3**	9.3		60	135	50	150	50
2,4-Dinitrotoluene	0.08	0.0199	0.0118	**0.020**	0.1	**0.3**	15.1		60	135	50	145	50
2-Nitrotoluene	0.08	0.0133	0.0171	**0.017**	0.1	**0.3**	17.5		45	135	30	150	50
4-Nitrotoluene	0.08	0.0180	0.0171	**0.018**	0.1	**0.3**	16.6		50	130	35	145	50
3-Nitrotoluene	0.08	0.0238	0.0460	**0.046**	0.1	**0.3**	6.5		50	130	35	145	50
				Mean MQL/MDL ratio:			11.6						

Criteria for the ratio (RD) of mean amount found to MDL: >1 but <5 for water.

Mean observed ratio: 4.2 on C-18 column and 3.9 on C8/CN column.

Number of ratio outliers: 5 out of 14 on C-18 and 3 out of 14 on C8/CN column.

Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.

Control and marginal exceedance (ME) limits for LCS recoveries (%R) are from DoD QSM, Final Version 2, June 2002.

DoD QSM Control and marginal exceedance (ME) limits for LCS recoveries (%R) were also applied to MS recoveries.
Limits for RPD are from the USACE EM200-1-3, Appendix I, I Feb 2001.

The following criteria will apply for marginal exceedances:

Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).

NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.

Table V
Soil/Sediment Sample MDL, LLR, MQL and QC Limits for LCS and MS
Standard 8330 List of Compounds

Nitroaromatic and Nitramine Explosives Analysis by HPLC

Analytical Management Laboratories

Preparation Method:	8330A						HPLC Analysis Primary Column					C-18	
Analysis Method:	8330A		Matrix:		Soil		Confirmation Tandem Column:					C8/CN	
	MDL	**MDL**	**MDL**	**MDL**	**LLR**	**MQL**	**Ratio (RQ)**		**DoD QSM LCS & MS Control Limits**		**DoD QSM LCS & MS ME Limits**		**RPD**
Compound	Spike	C-18	C8/CN	Report	Report	Report	QL/DL	flag	Lower	Upper	Lower	Upper	Limit
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg			**%R**	**%R**	**%R**	**%R**	%
HMX	0.3	0.0798	0.0807	**0.081**	0.1	**0.3**	3.7		75	125	65	135	50
RDX	0.3	0.0610	0.0725	**0.073**	0.1	**0.3**	4.1		70	135	65	145	50
1,3,5-Trinitrobenzene	0.3	0.0130	0.0309	**0.031**	0.1	**0.3**	9.7		75	125	65	135	50
1,3-Dinitrobenzene	0.3	0.0116	0.0321	**0.032**	0.1	**0.3**	9.3		80	125	70	135	50
Tetryl	0.3	0.0243	0.0519	**0.052**	0.1	**0.3**	5.8		10	150	0	172	50
Nitrobenzene	0.3	0.0194	0.0603	**0.060**	0.1	**0.3**	5.0		75	125	70	130	50
2,4,6-Trinitrotoluene	0.3	0.0177	0.0415	**0.041**	0.1	**0.3**	7.2		55	140	45	155	50
4-Amino-2,6-dinitrotoluene	0.3	0.0352	0.0542	**0.054**	0.1	**0.3**	5.5		80	125	75	130	50
2-Amino-4,6-dinitrotoluene	0.3	0.0211	0.0498	**0.050**	0.1	**0.3**	6.0		80	125	75	130	50
2,6-Dinitrotoluene	0.3	0.0239	0.0600	**0.060**	0.1	**0.3**	5.0		80	120	70	130	50
2,4-Dinitrotoluene	0.3	0.0140	0.0493	**0.049**	0.1	**0.3**	6.1		80	125	75	130	50
2-Nitrotoluene	0.3	0.0391	0.0716	**0.072**	0.1	**0.3**	4.2		80	125	70	130	50
4-Nitrotoluene	0.3	0.0398	0.0562	**0.056**	0.1	**0.3**	5.3		75	125	70	135	50
3-Nitrotoluene	0.3	0.0131	0.0460	**0.046**	0.1	**0.3**	6.5		75	120	70	130	50
				Mean MQL/MDL ratio:			6.0						

Due to sensitivity differences in the HPLC columns the criteria for the ratio (RD) may need to be expanded to values >10.

Criteria for the ratio (RD) of mean amount found to MDL: >1 but <10 for soil.

Mean observed ratio: 14.5 on C-18 column and 7.3 on C8/CN column.

Number of ratio outliers: 9 out of 14 on C-18 and 2 out of 14 on C8/CN column.

Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.

Control and marginal exceedance (ME) limits for LCS recoveries (%R) are from DoD QSM, Final Version 2, June 2002.

DoD QSM Control and marginal exceedance (ME) limits for LCS recoveries (%R) were also applied to MS recoveries.
Limits for RPD are from the USACE EM200-1-3, Appendix I, I Feb 2001.

The following criteria will apply for marginal exceedances:

Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).

NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.

Table VI
Aqueous Sample MDL, LLR, MQL and QC Limits for LCS and MS
TNT Metabolites

Metabolites of Nitroaromatic and Nitramine Explosives Analysis by HPLC

Analytical Management Laboratories

Preparation Method:	3535A						HPLC Analysis Primary Column					C-18	
Analysis Method:	8330A		Matrix:		Water		Confirmation Tandem Column:					C8/CN	
	MDL	**MDL**	**MDL**	**MDL**	**LLR**	**MQL**	**Ratio (RQ)**		**LCS Limits**		**MS Limits**		**RPD**
Compound	Spike	C-18	C8/CN	Report	Report	Report	QL/DL	Flag	Lower	Upper	Lower	Upper	Limit
	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L			**%R**	**%R**	**%R**	**%R**	%
2,6-Diamino-4-nitrotoluene	0.07	0.006	0.013	0.013	0.023	**0.075**	5.8		70	130	50	140	50
2,4-Diamino-6-nitrotoluene	0.14	0.017	0.035	0.035	0.047	**0.15**	4.3		70	130	50	140	50
2-Methy-3-nitroaniline	0.3	0.015	0.030	0.030	0.10	**0.6**	20		70	130	50	140	50
4-Methy-3-nitroaniline	0.3	0.010	0.026	0.026	0.10	**0.6**	23		70	130	50	140	50
2-Methy-5-nitroaniline	0.07	0.004	0.012	0.012	0.013	**0.075**	6.2		45	140	45	140	50

LCS and MS %R limits and sporadic marginal failure limits for these special analytes were adopted from the USACE EM200-1-3, Appendix I, I Feb 2001. Guidance from DoD QSM was unavailable.

Sporadic marginal failure (SMF) limits for LCS and MS recoveries are 40% to 150%.
Sporadic marginal failure (SMF) limit for LCS, MS or sample duplicates is 60%.

Table VII
Soil/Sediment Sample MDL, LLR, MQL and QC Limits for LCS and MS
Nitroglycerin and PETN

Nitroglycerin and PETN Analysis by HPLC

Analytical Management Laboratories

Preparation Method:	8332/8330A						HPLC Analysis Primary Column					C-18	
Analysis Method:	8332/8330A		Matrix:		Soil		Confirmation Tandem Column:					C8/CN	
	MDL	**MDL**	**MDL**	**MDL**	**LLR**	**MQL**	**Ratio (RQ)**		**LCS Limits**		**MS Limits**		**RPD**
Compound	Spike	C-18	C8/CN	Report	Report	Report	QL/DL	Flag	Lower	Upper	Lower	Upper	Limit
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg			**%R**	**%R**	**%R**	**%R**	%
Nitroglycerin (NG)	5.0	0.142	2.488	2.49	2.5	**10.0**	4.0		70	130	50	140	50
Pentaerythritol tetranitrate (PETN)	5.0	0.119	1.863	1.86	2.5	**10.0**	5.4		70	130	50	140	50

LCS and MS %R limits and sporadic marginal failure limits for these special analytes were adopted from the USACE EM200-1-3, Appendix I, I Feb 2001. Guidance from DoD QSM was unavailable.

Sporadic marginal failure (SMF) limits for LCS and MS recoveries are 40% to 150%.
Sporadic marginal failure (SMF) limit for LCS, MS or sample duplicates is 60%.

Table VIII
Soil/Sediment Sample MDL, LLR, MQL and QC Limits for LCS and MS
Nitroguanidine

Nitroguanidine Analysis by HPLC

Analytical Management Laboratories

Preparation Method:	8330A						HPLC Analysis Primary Column					C-18	
Analysis Method:	8330A		Matrix:		Soil		Confirmation Tandem Column:					C8/CN	
	MDL	**MDL**	**MDL**	**MDL**	**LLR**	**MQL**	**Ratio (RQ)**		**LCS Limits**		**MS Limits**		**RPD**
Compound	Spike	C-18	C8/CN	Report	Report	Report	QL/DL	Flag	Lower	Upper	Lower	Upper	Limit
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg			**%R**	**%R**	**%R**	**%R**	%
Nitroguanidine (NQ)	0.191	0.0101	NA	0.010	0.1	**0.3**	30		70	130	50	140	50

LCS and MS %R limits and sporadic marginal failure limits for these special analytes were adopted from the USACE EM200-1-3, Appendix I, I Feb 2001. Guidance from DoD QSM was unavailable.

Sporadic marginal failure (SMF) limits for LCS and MS recoveries are 40% to 150%.
Sporadic marginal failure (SMF) limit for LCS, MS or sample duplicates is 60%.

Table IX
List of Appendices

Appendix Number	Appendix Topic
1	Stock Standards, Concentrations and Typical Expiry Dates
2	Initial Calibration (ICAL) Summary (Form-6 equivalents) Instrument Printouts and Excel Spreadsheets
3	Calibration Verification (ICV and CCV) Summary (Form-7 equivalents) Instrument Printouts and Excel Spreadsheets
4	Standard Chromatograms for CCV (LC-18 Column) Instrument Printouts
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6	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
7	Sample Result Worksheets (Form-10 equivalents)
8	USACE Method Quality Objectives for Method 8330 AFCEE MQOs for Method 8330 DoD QSM (Final Revision 2) Method Quality Objectives
9	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents) Miscellaneous MS Information
10	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents) Miscellaneous LCS information
11	Surrogate Recovery Reports (Form-2 equivalents)
12	Method Detection Limit (MDL) Studies Summary
13	QC Association Forms (Form-4 equivalents) Sample Preparation Logsheets Instrument Run Log
14	Sample Reports (Form-1 equivalents)

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. O-PAH-HPLC-RC

POLYNUCLEAR AROMATIC HYDROCARBONS (PAHs or PNAs) by HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (EPA 550.1 and SW846-8310)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

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Laboratory Director, Analytical Management Laboratories Date

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Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

POLYNUCLEAR AROMATIC HYDROCARBONS (PAHs or PNAs) by HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (EPA 550.1 and SW846-8310)

1.0 SCOPE AND APPLICATION

1.1 This SOP, which is based on EPA 550.1 and SW-846 Method 8310, is used to determine the trace levels of the following 16 polynuclear aromatic hydrocarbons (PAH) in ground water, wastes and soil by High Pressure Liquid Chromatography (HPLC) and Ultra Violet light and/or a fluorescence detector. All sample analyzed require extraction procedures listed in this standard operating procedures document.

Acenapthene	Chrysene
Acenapthylene	Dibenzo (a, h) anthracene
Anthracene	Fluoranthene
Benzo (a) anthracene	Fluorene
Benzo (a) pyrene	Indeno(c, d) pyrene
Benzo (b) fluoranthene	Napthalene
Benzo (ghi) perylene	Phenanthrane
Benzo (k) fluoranthene	Pyrene

1.2 Method detection limits (MDL), sensitivity and optimum ranges vary with matrices. MDL data are listed in Table 1 (water) and Table 2 (soil). Since the sensitivity of this method usually depends on the level of interferences rather than instrumental limitations, samples extracts are placed through a silica gel cleanup step prior to analysis. When interferences cannot be removed, samples are diluted for analysis, which usually raises the detection limits.

2.0 SUMMARY OF METHOD

2.1 Aqueous samples are extracted using either a separatory funnel method or by solid phase extraction procedure:

2.1.1 Methylene Chloride is used as the solvent for separatory funnel extraction and the sample extracts are exchanged with acetonitrile during the final extraction step.

2.1.2 Solid phase extraction employs C-18 cartridges.

2.2 Soil samples are extracted by sonication or soxhlet.

2.3 When a sample contains oily material or is highly colored (dark amber), a silica gel clean up procedure is required prior to the final concentration step. When a sample requires the clean up step, all associated blanks and spikes are placed through the clean up procedures as well.

2.4 This SOP utilizes a reverse phase HPLC system containing a UV absorbance detector and a fluorescence detector.

2.4.1 The UV detector operates at a wavelength of 254 nm. The fluorescence detector operates at an excitation wavelength of 280-nm and emission wavelength of 490 nm.

2.4.2 The sample extracts are injected using an autosampler into the mobile phase stream. The mixture of sample extract and mobile phase are forced into a packed column having a LC-PAH stationary phase.

2.4.3 The analyte absorbance or emission is recorded electronically using a chromatography data system. The chromatographic peaks are separated by the retention time order of the analytes.

3.0 INTERFERENCES

3.1 Sample extracts may contain artifacts, which are matrix related or are present as result of the laboratory preparation process. To monitor contaminants, which may be introduced during sample preparation, a method blank is extracted along with each sample batch. Interferences, which may be caused by the sample matrix, are reduced by cleaning up sample extracts using a silica gel cleanup procedure.

3.2 Non target analytes such as substituted PAHs may interfere with target chromatographic peaks; these are reduced by using the method recommended column (Supelco LC-PAH 250 mm X 4.6 mm) and the recommended mobile phase gradient program.

3.3 For a positive confirmation of target PAHs samples are analyzed on a mass spectrometer coupled by a particle beam interface to a HPLC. This procedure is called Particle Beam LC/MS analysis for PAHs. The sensitivity of this method is 1 – 2 ng/uL in extracts.

4.0 APPRATUS AND MATERIALS

4.1 Extraction Apparatus: K-D (Kuderna-Danish)

4.1.1 Concentrator tube: 10 ml

4.1.2 Evaporation Flask: 500 mL

4.1.3 Snyder column: Three ball macro

4.2 Boiling Chips: Teflon, rinsed with 1,2-Dichloromethane

4.3 Water Bath: Heated to boil 1,2-Dichloromethane

4.4 Syringe: Hamilton; 0.1 mL; 1mL; 5 mL; 10mL

4.5 HPLC system

4.5.1 HPLC: Liquid Chromatographs (HP1090) equipped with tunable absorbance detectors, A/D converters, circulating cooling bath, and a Chemstation software for LC and LC/MS systems. Two complete additional HPLC units (Agilent 1100) that are used for explosives analysis are also available as back-up equipment for PAHs analysis.

4.5.2 HP1090 gradient pump controller.

4.5.3 HPLC columns with comparable performance characteristics are available from a number of sources (Phenomenex, Waters, Supelco, etc.). Actual column in use at any given time is subject to change. The column that is currently in use is Supelcosil LC-PAH 250 mm X 4.6 mm, 5 um.

4.5.4 UV detectors (Waters Model 486) are capable of achieving a 0.001 AUFS sensitivity range at 254-nm and 220-nm wavelengths.

4.5.5 Fluoroscence detector - LDC Analytical 4100 (Excitation 280 nm; Emission 410 nm)

4.5.6 Analog to digital (A/D) converters (Agilent Model 35900E), which are attached to the Agilent Chemstation.

4.5.7 Chemstation for data acquisition and processing (Agilent Rev. A.08.03 (847)).

4.5.8 Laser printer(Cannon LBP-860 or equivalent)

4.6 Circulating cooling bath (NESLAB RTE-111) capable of temperature control in the 0°C to 50°C.

4.7 Labline vortexer (various models).

4.8 Analytical balance (Leco LB-8 or equivalent) with a precision of ± 0.0001 g for gravimetric measurements.

4.9 Corning stir plate (Various models).

4.10 Volumetric Flasks, Class A : 5 mL; 10 mL; 25 mL; 50 mL; 100 mL

4.11 SPE extraction equipment:

4.11.1 Two SPE manifolds (Supelco 12-positions and 24-positions) for sample extraction, vacuum control and reservoir for spent water sample collection (Kontes Ultraware).

4.11.2 Vacuum pump (Gast Model 2Z866)

4.11.3 Supelco SPE extraction cartridges with C-18 phase; Waters Porapak® R_{DX} C-18 SPE cartridge (Part # WAT047220, reserve lot # W1023B3 or equivalent for one-year supply)

4.11.4 Sep-Pak adapter for Waters Porapak® R_{DX} C-18 SPE cartridges

4.11.5 Sep-Pak reservoirs for Waters Porapak® R_{DX} C-18 SPE cartridges

4.12 Gelman Science 0.45 um Teflon filter, 47 mm diameter

4.13 Hamilton syringes- various sizes

4.14 Whatman filtration and degassing unit with 2 liter flask

4.15 HPLC vials (1.5-ml) from Agilent

4.16 Culture tubes, 13 x 100 mm (Fisher)

4.17 Eppendorf pipettes, 100-1000 µL and 10-100 µL

5.0 REAGENTS

5.1 All chemicals used are HPLC grade or better. If contamination is found at or above method detection limit, all suspected chemicals are screened individually and replaced.

5.2 Reagent water: Type I water is produced at the laboratory using USFilter water system equipped with a 0.45 micron filter. See AML SOP on reagent water for details.

5.3 Solvents

5.3.1 Acetonitrile- HPLC grade (EM Science)

5.3.2 Methanol – HPLC grade (EM Science)

5.4 Stock Standard Solutions (SSS). Stock solutions expire on the dates shown by the vendor, which is generally 1 to 3 years from the date of manufacture. Please note that the expiration date may strictly apply if the container is not opened and stored under manufacturer's recommended conditions (freezer). Do not break the seal if these solutions are not used immediately to prepare other standard solutions. Transfer stock solutions immediately after opening the ampules or vials to Teflon®-lined screw cap vials and store them in accordance with manufacturer instructions, if any. The stock solutions should be stored in a freezer. Replace standards after 1 year or sooner if signs of degradation (unknown peaks, concentration changes, etc.) are observed. Stock solutions are used to prepare intermediate solutions.

5.4.1 Primary Stock Standard (PSS) Solution for ICAL, CCV, LCS, and MS - A Combined Primary Stock Standard Solution (see Appendix 1 for details) containing all 16-target compounds listed in section 1.1 at varying concentrations (20 to 1000-µg/mL) is purchased from Ultra Scientific (Catalog No. PM-831A).

5.4.2 Second Source Stock Standard (SSS) Solution for ICV - The PSS solution (see Appendix 1 for details) containing all 16 target compounds is obtained from Supelco (Catalogue No. 49156).

5.4.3 Surrogate Stock Solution: Either decafluorobiphenyl (DFBP) or the compound, 1-Chloro-3-Nitrobenene (CNTB) can be used as the surrogate.

5.4.3.1 A Stock Solution (545-µg/mL) of 1-Chloro-3-Nitrobenzene is prepared by weighing 0.0545-g of the neat material (Aldrich, catalog # 218758) and making the solution up to 100-mL with methanol. Store this solution in a freezer. Discard Surrogate Stock Solution 2-years from the date of preparation or sooner if signs of degradation (unknown peaks in the MB, concentration changes, etc.) are observed.

5.4.3.2 A stock standard solution of decafluorobiphenyl (1000-µg/mL) is purchased from Ultra (Catalogue No. IST-151).

5.4.3.3 ***Note – The analyst may choose a different surrogate if the surrogate interferes with target compounds that may be added to this method.***

5.5 Surrogate Sample Spike Solution

5.5.1 CNTB- The CNTB surrogate sample spike solution (2000-ng/mL) is prepared by diluting 0.734-ml of the stock solution to 100-mL with methanol. Use 1.0-mL of the Surrogate Sample Spike Solution to spike each aqueous sample (normally 1-L) including all quality control (QC) samples (MB, LCS, MS). Use 1-mL of this solution to spike soil samples (normally 30-g) including all associated QC samples.

5.5.2 DFBP- The DFBP surrogate sample spike solution (2000-ng/mL) is prepared by diluting 0.2-mL of the stock solution to 100-mL with methanol. Use 1.0-mL of the Surrogate Sample Spike Solution to spike aqueous and soil samples.

5.6 ICAL Solutions - These are prepared daily from the primary stock solution (PSS). Initial Calibration (ICAL) Standards. Five ICAL solutions are prepared by diluting aliquots of the PSS (Section 5.4.1) to 1-mL with acetonitrile. Add 1-mL of acetonitrile to six HPLC injection vials. Remove volumes of the solvent equivalent to the volume of the PSS that will be added as described below.

5.6.1 STD-0 is the calibration blank. This standard contains neat acetonitrile.

5.6.2 STD-1 is prepared by diluting10-µL of the PSS to 1-mL.

5.6.3 STD-2 is prepared by diluting 20-µL of the PSS to 1-mL.

5.6.4 STD-3 is prepared by diluting 50-µL of the PSS to 1-mL.

5.6.5 STD-4 is prepared by diluting 75-µL of the PSS to 1-mL.

5.6.6 STD-5 is prepared by diluting100-µL of the PSS to 1-mL.

5.7 CCV Solution - This is prepared as described in Section 5.6.4

5.8 ICV Solution - This is also prepared as described in Section 5.6.4 using a second source standard (Section 5.4.2).

5.9 LCS/MS Spiking Solution: Dilute 25-mL of the PSS (Section 5.4.1) to 100-mL with methanol. Spike 1-mL of this solution to each LCS or MS sample. The extract sample (5-mL final volume) concentration will equal ICV and CCV concentrations.

5.10 Method Blank (MB)

5.10.1 Aqueous Sample - Use 1-L of reagent Water for both separatory funnel and SPE extraction methods.

5.10.2 Soil/Sediment Samples - Use an amount of anhydrous sodium sulfate that equals the amount of sample extracted (10-g for Soxhlet and 30-g for sonication).

5.11 Laboratory Control Samples (LCS and LCSD) - These are prepared as shown above for MB (Section 5.10) with the exception that they are spiked with 1-mL of the spiking solution (Section 5.9) prior to extraction.

5.12 Matrix Spike Samples (MS and MSD) - These are prepared by spiking actual samples designated for MS/MSD analysis with 1-mL of the spiking solution (Section 5.9) prior to analysis.

5.13 Mobile Phase: Mobile phase for HPLC analyses are prepared by filtering the solvent through a 0.45-µm filter and degassing them with helium prior to use. Prepare the following solvents for use in chromatography:

5.13.1 HPLC grade water (Type I)

5.13.2 HPLC grade acetonitrile (ACN)

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Aqueous Samples - One liter amber glass bottles with Teflon lined screw caps may be used for collecting aqueous samples in accordance with EPA SW-846 method 8310.

6.1.1 Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.).

6.1.2 Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples.

6.1.3 Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.

6.1.4 Water samples must be extracted within 7 days of collection. Samples extracts are stored in amber bottles and/or vials at 4 degrees Celsius just prior to analysis.

6.1.5 Sample extracts should be analyzed on the instruments within 40 days from the date of extraction.

6.2 Soil, Sediment or Waste Samples - One 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples in accordance with EPA SW-846 method 8310.

6.2.1 Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each solid sample.

6.2.2 Quality control samples such as MS and MSD should be treated as regular environmental samples.

6.2.3 Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.

6.2.4 These samples may be shared with samples collected for other analyses such as semivolatiles (8270C), PCBs (8082), Diesel (DRO, 8015-mod.) and pesticides (8081, 8140, etc.).

6.2.5 Soil and sediment samples must be extracted within 14 days of collection. Samples extracts are stored in amber bottles and/or vials at 4 degrees Celsius just prior to analysis.

6.2.6 Sample extracts should be analyzed on the instruments within 40 days from the date of extraction.

6.3 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Analytical data generated outside of the specified holding times must be considered to be minimum values.

7.0 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control contain a number of regulated compounds at high concentrations. A number of polynuclear aromatic hydrocarbons are human carcinogens (e.g. benzo (a) pyrene) that are regulated by the Safe Drinking Water Act (SDWA). The toxicity of the PAH compounds present in complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 Proper sample disposal practices (see AML Waste Disposal SOP) should be followed to minimize exposure to toxic compounds.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Aqueous and soil samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters after the solvent(s) have been allowed to evaporate in a hood.

8.4 Return soil samples containing hazardous compounds (e.g., dioxins, explosives) to the client for proper disposal.

9.0 SAMPLE EXTRACTION PROCEDURE

A sample batch may not exceed twenty (20) environmental samples in addition to the method blank (MB), laboratory control sample (LCS) and a LCS duplicate (LCSD, which is optional). Matrix spike (MS) and MS duplicate (MSD) samples should be counted as environmental samples. Document sample weights, volumes, extraction QC batch number, lot number for solvents and reagents, extract volumes, date, analyst initials, etc. in the sample preparation log book. Two separate extraction procedures are described for water and soil/sediment samples.

9.1 Extraction of Aqueous Samples by Separatory Funnel Method

9.1.1 Samples should be at room temperature. Allow them to warm up to room temperature. The pH of the water samples should be between 6.5 and 7.5. Measure using a pH paper and record it in the logbook. This is accomplished by dipping the tip of a Pasteur pipette into the water and placing the droplets of water on the pH paper. Never place the pH paper into the water sample. Adjust pH with 50 % H_2SO_4 or NaOH.

9.1.2 Using a black marker, mark the meniscus of the water in the sample container. Pour the entire contents of the container into a 2-liter separatory funnel. Rinse the sample container with 3 aliquots of 20-mL methylene chloride and place the rinse in the separatory funnel.

9.1.3 Add distilled water to the sample container up to the marked meniscus. Pour the distilled water into a 1-liter graduated cylinder. Record this measured volume in the laboratory logbook.

9.1.4 Prepare MB, LCS, LCSD, MS, and MSD samples as described previously.

9.1.5 Add 1-mL of surrogate solution to all samples (includes QC samples such as MB, LCS, LCSD, MS and MSD).

9.1.6 Add 60-mLs of methylene chloride to the separatory funnel and shake for 2 minutes. Take care to vent the separatory funnel to relieve excess pressure. Allow the organic layer to separate from the water layer for a minimum of 10 minutes.

9.1.7 Prepare a funnel (plugged with glass wool and covered by sodium sulfate. Rinse Sodium Sulfate bed with 5 to 10 mL methylene chloride. Discard the washings. Place the funnel inside a K-D apparatus fitted with 10 mL K-D tube.

9.1.8 Drain the methylene chloride extract (bottom layer) through the funnel into the K-D flask.

9.1.9 Repeat the extraction 3 times using 60-mL of methylene chloride (Section 9.1.5 and 9.1.7).

9.1.10 Add two PTFE boiling chips to the K-D flask and attach a three ball Snyder column. Wet the Snyder column with 1 mL of methylene chloride. Place the apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed in vapor. The balls of the column will actively chatter but the chamber should not flood.

9.1.11 When the extract reaches approximately 1-ml to 10-ml, add 5-ml of acetonitrile and allow sample to come back down to less than 3-mL.

9.1.12 Remove the K-D flask and allow cooling for at least 10 minutes. Place the K-D tube in a N-Evap unit and evaporate off any residual amount of dichloromethane. Adjust the final volume to 1-mL using fresh acetonitrile.

9.1.13 Filter the extract through a 0.45-micron Teflon filter disk and collect approximately it in an Agilent autosampler vials for HPLC analysis.

9.2 Extraction of Aqueous Samples by SPE Method

9.2.1 Perform extractions using Supelco SPE vacuum manifold. Condition the SPE tube with 2 to 5-ml of methanol, followed by 2-ml of tap water (NOTE: Do NOT use distilled water or higher grade water since the aqueous conditioner should be similar to the sample in pH, salt concentration, etc.). The tube should not dry up anytime during the conditioning procedure.

9.2.2 Mark the meniscus on the bottle with a marker. Prepare the water samples (see Section 9.1.1) and associated QC samples (see Section) as described previously.

9.2.3 Disconnect vacuum source before setting up sample extraction. Add 1-mL of the surrogate sample spike solution and mix it thoroughly by gently swirling the bottle.

9.2.4 Dip one end of the sample drain tube into the bottom of the sample bottle and snap the other end into the SPE cartridge. Adjust the flow rate of the sample through the SPE cartridge to less than 10-ml/minute by adjusting the vacuum controller for each sample in the manifold.

9.2.5 After all the samples have passed through the cartridge, but before the SPE tube goes dry, remove the connecting tube from the cartridge and elute the target compounds with 5.0-mL acetonitrile. Pulse the vacuum pump to start the flow, then let the solvent drip through under gravity alone at a flow rate of approximately 1-mL/minute. Collect the acetonitrile extract in clean 10-mL screw-cap tubes and adjust final volume to 5-mL before instrumental analysis.

9.2.6 Filter approximately 3-mL through a 0.45 micron Teflon filter disk; discard the first 1 to 2-mL and collect approximately 1- to 1.5-mL in Agilent autosampler vials for HPLC analysis.

9.2.7 Add distilled water to the empty sample container up to the marked meniscus. Pour the distilled water into a 1-liter graduated cylinder. Record this measured volume in the logbook.

9.3 Extraction of Soil and Sediment Samples by Sonication

9.3.1 Thoroughly mix sample as defined in the AML SOP on subsampling of containers. Since sample results are reported on dry weight basis, determine percent solids as described below. Weigh out 5-g of sample into an aluminum weight boat to three significant digits. Place sample in a 104°C oven for 24 hours for percent moisture determination. See applicable SOP for percent solids determination. If percent solids is determined by another group (e.g. metals analysis section), this procedure need not be repeated.

9.3.2 Weigh out 30 grams of sample into a 500-mL beaker. Record weight to three significant digits into logbook.

9.3.3 Prepare associated QC samples (MB, LCS, LCSD, MS, and MSD) as described previously.

9.3.4 Add 1-mL of surrogate solution to all samples (includes QC samples such as MB, LCS, LCSD, MS and MSD).

9.3.5 Add 60-g of sodium sulfate to sample to each sample. Additional sodium sulfate may need to be added to sediment and other wet soil samples to dry them completely. Mix well using a spoon.

9.3.6 Add 60-mL dichloromethane to the sample beaker. After cleaning by rinsing the sonic horn with dichloromethane place it into sample beaker.

9.3.7 Sonicate for 3 minutes at full power with the mode switch set at pulse (on 50% of the time). Prepare filter funnel and K-D equipment as described in Section 9.1.7 and collect filtered sample extract in the K-D.

9.3.8 Repeat twice (Section 9.3.6 and 9.3.7) more with fresh 60-mL aliquots of methylene chloride. contents as per instruction in sections. Decant the solution in to the same K-D as above.

9.3.9 Remove methylene chloride and complete solvent exchange as described in Section 9.1.10 to 9.1.13.

9.4 Extraction of Soil and Sediment Samples by Automated Soxhlet (IKA or Soxtec) - See other determinative procedures (e.g. AML SOP on 8081-organichlorine pesticides) for more details on soil sample preparation using the automated Soxhlet.

9.4.1 Characterize soil or sediment sample as described in Section 9.4.1.

9.4.2 Weight out 10-g (30-g for IKA) of soil. Dry it with 20-g (60-g for IKA) sodium sulfate. Add the entire contents into a fiber glass thimble and place thimble onto a Soxtec unit.

9.4.3 Add 60-mL of dichloromethane and set temperature program to methylene chloride (NOTE: Use program 1 or follow instructions in the manual).

9.4.4 The water recirculator should be set a temperature less than 4°C. Let solvent recirculate for 18 hours.

9.4.5 Collect the extract and perform volume reduction and solvent exchange as described in Section 9.1.10 to 9.1.13.

9.5 Extract Cleanup Procedure (Silica Gel) - Perform cleanup on extracts immediately after final extract volume adjustment (Section 9.1.12 or 9.2.5) but prior to filtration through 0.45-µm filters.

9.5.1 Pack a 50-mL Champaign tube with heat conditioned Silica Gel. Add a stopcock to the tip.

9.5.2 Add 25 to 30-mL of dichlormethane to the column reservoir. When the top of rinse solvent reaches the top of the silica gel, close the stopcock.

9.5.3 Add sample and elute at a rate of 1 drop per second. Rinse with one column volume of dichloromethane. Discard the washings.

9.5.4 Add 30-mL of a 20% Ether in dichloromethane to elute the PAHs from the column. Collect this fraction in an Erlenmeyer flask.

9.5.5 Repeat step 9.5.4 twice and collect them in the same flask.

9.5.6 Perform volume reduction, solvent exchange and filtration steps as described in Sections 9.1.0 to 9.1.13).

10.0INSTRUMENTAL (HPLC) ANALYSIS

10.1 Chromatography Conditions - Follow the instrument manufacturer guidelines and software manuals to set up HPLC to good operating conditions. The C-18 column is used as the primary column for analysis. Set chromatographic conditions for the PAH analysis as describe below:

10.1.1 Gradient Program: Use the following mobile phase gradient (filter with 0.45 um filter then degas with Helium 20 minutes before using; let mobile phase stand for at least 30 min).

Time (min.)	Acetonitrile %	Water %
0.0	60	40
5.0	60	40
30	100	0
45	100	0
48	60	40
50	60	40

10.1.2 Pump Flow rate (Initial conditions) - Set Pump A (Acetonitrile) at 1.5-mL/min. and Pump B (Water) at 1.5 mL/min.

10.1.3 Detectors: Detector A (UV) set wavelength at 254-nm and sensitivity (AUFS) at 0.05; Detector B (Fluorescence) set emission at 410-nm and excitation at 280-nm.

10.1.4 Autosampler - Injection Volume: 100 uL; Mode: Auto; and Analysis time: 50 min

10.1.5 Data Collection:
Method #: 8310 (stop time = 50 min; peak area mode; calibration Response factor)
Calibration: Multiple concentrations (5 points or more). Edit Previous sequence and rename by using new date of collection MMDDYY
Save sequence and data files under subdirectory: DATA1 or DATA2.

10.1.6 The following is a typical injection sequence that involves the generation of an initial calibration curve (ICAL). Begin and end sample analysis with a CCV. Analyze one CCV after every 10 injections.

1. Calibration/Reagent Blank
2. ICAL STD 1
3. ICAL STD 2
4. ICAL STD 3
5. ICAL STD 4
6. ICAL STD 5
7. Reagent Blank
8. ICV
9. ICB/CCB
10. CCV (#1)
11. Method Blank (MB)
12. Laboratory Control Sample (LCS)
13. LCS Duplicate (LCSD)
14. Matrix Spike (MS)
15. Matrix Spike Duplicate (MSD)
16. CCV (#2)
17. Sample # A1 (used for MS)
18. Sample # A2
19. Sample # A3

20. Sample # A4
21. Sample # A5
22. Sample #A6
23. Sample #A7
24. Sample #A8
25. Sample #A9
26. Sample #A10
27. CCV (#3)

(28 to 37) Sample # A11 to A20
(38) CCV (#4)

10.2 Calibration

10.2.1 Set up the analysis sequence containing a minimum of five calibration solutions for initial calibration (ICAL) followed by the analysis of a second source ICV solution. Include calibration blanks as appropriate before and after the analysis of ICAL and ICV solutions. Summarize initial calibration data in Form-6 equivalents (see Appendix 2 for details). Using peak areas (or peak heights when appropriate), calculate calibration factors (CF) as described in Section 11.1, the mean calibration factor as described in Section 11.2, standard deviation and percent relative standard deviation (RSD) as described in Section 11.3 for each compound.

10.2.2 The %RSD for each compound should be 20% or less. The maximum allowable %RSD for any target analyte is 40% for USACE projects and 30% for AFCEE projects. The maximum allowable %RSD for project-specific contaminants of concern (COC) is 20%. Check with the Project Manager for a list of COCs for the specific project. If the %RSD for any non-COC compound is >20%, calculate the mean of the %RSDs of <u>all</u> target compounds in the calibration solution. If the mean %RSD is <20%, the initial calibration employed is acceptable for all compounds except those with RSD >20%. For compounds with RSD >20%, use linear regression method. The correlation coefficient for linear regression (r^2) should be >0.990 according to SW-846. It should be >0.995 for USACE and AFCEE projects. The coefficient of determination (COD) for non-linear regression (requires a minimum of 6 calibration points) should be >0.990. Correct the problem and reanalyze ICAL solutions if the %RSD and/or correlation coefficient are not met for any target compound. Use mean calibration factors for calculation of sample amounts. See SW-846 Method 8000B and/or USACE documents if additional guidance is needed.

10.2.3 An initial verification (ICV) is performed before any sample extracts are analyzed using the ICAL. For USACE and Air Force (see AFCEE QAPP) projects, a second source ICV different from the initial calibration standard should be run immediately after the initial calibration to find any potential systematic errors on calibration standards. Afterwards, continuing calibration verification (CCVs) solutions of the same source is analyzed at the beginning, end, and after every ten samples of an analytical batch. Calculate calibration factor (CF) for each compound as described in Section 11.1 and calculate percent difference (%D) between the CF for ICV and the mean CF from the ICAL as described in Section 11.4. Summarize calibration verification in Form-7 equivalents (see Appendix 3 for details).

10.2.4 The %D for ICV should be within ±15% (%Rec. = 85% to 115%) for each compound according to SW-846, USACE and AFCEE. Contact your supervisor (see Appendix 8 for details) on USACE and AFCEE requirements.

10.2.5 Continuing calibration verification (CCV) is performed at the beginning of sample analysis, after every 10 samples and at the end of run using the same source standard as ICAL. Calculate calibration factors for the daily standard and the %difference (%D) of each from the mean calibration factor from the initial calibration as described in Section 10.2.4 for ICV. Summarize calibration verification in Form-7 equivalents (see Appendix 3 for details).

10.2.6 The %D for each compound in CCV should be 15% or less for each compound. The %D should not exceed 30% for any one compound (USACE criteria). If the %D for any compound is >15%, calculate the mean of the %Ds for <u>all</u> target compounds in the calibration solution. If the mean %D is <15%, the initial calibration employed is acceptable. If the mean %D is >15%, restore instrument to conditions similar to those used for initial calibration and repeat CCV. If CCV fails again, perform an initial calibration as described in the previous section. For USACE projects, apply the SMF criteria shown in Section 10.2.4. The number of compounds with sporadic marginal failure (SMF) that can exceed the 15% criteria for USACE projects is as follows: none if less than 7 PAH compounds are reported from the analysis; 1 compound if between 7 and 15 are reported; and 2 compounds if >15 are reported. The number of SMF compounds is zero for AFCEE projects. Compounds with %D >15% may have greater uncertainty, which may require data qualification.

10.3 Sample Analysis

10.3.1 Set up the following sequence for sample analysis: CCV, MB, LCS, LCSD, MS, MSD, CCV, sample used for MS, 10 samples (including the sample used for MS), and CCV. Repeat CCV and 10 samples and end the run with a CCV.

10.3.2 Analyze samples on the HPLC and calculate sample amounts using mean calibration factors from the initial calibration. Perform calculations as described in Section 11.7 for water samples and Section 11.8 for soil samples. Document analysis on instrument printouts consisting of quantitation reports and chromatograms in project files (see Appendix 4 and 5 for details).

10.3.3 For a compound to be reported as positive, a chromatographic peak must be present within the retention time (RT) window established for the compound.

10.3.4 If the concentration of any of the target compounds exceed the upper limit of calibration (ICAL), flag the data for affected compounds as estimated ("E" flag). The extracts should be diluted and reanalyzed until the concentrations are within the calibration limits for normal turnaround analyses. For rapid turnaround analyses sample results for compounds that are not contaminants of concern may be reported with "E" qualifiers if sufficient time is not unavailable for reanalysis. Check with Client Project Manager for project specific requirements.

10.3.5 The terms, method detection limit (MDL), reporting limit (RL) and method or practical quantitation limit (MQL or PQL) are defined in the AML SOP on MDL. These are summarized as they apply to the reporting of sample results:

10.3.5.1 MDL: Default MDLs for samples prepared and analyzed in strict accordance with default procedures. MDLs are shown in Table 1

and 2 for aqueous and soil samples. For actual samples, MDLs are corrected based on the deviation from default values. The corrected MDL value (sample detection limit or SDL) is the lowest amount that can be reported for the sample in question.

10.3.5.2 Lowest level for reporting (LLR or RL): The LLRs are cut-off limits that are defined in the AML SOP on MDLs. For multi-analyte methods, the cut-off limits are uniform round numbers that are equal to or greater than MDLs but less than the MQL. Under EPA guidelines, reporting limits are based on professional judgment and it can be equal to or greater than MDL but less than or equal to MQL. USACE is trying to standardize these cut-off limits at two times the MDL. For actual samples, LLRs are corrected based on the deviation from default values. The corrected LLR value is the lowest amount that can be reported for the sample (SRL) as present.

10.3.5.3 MQL: Default MQLs for samples prepared and analyzed in strict accordance with default procedures. For actual samples, MQLs are corrected based on the deviation from default values. The corrected MQL value is the lowest amount that can be reported for the sample (SQL) in question without qualifications as an estimated quantity (J flag). MQLs are normally based on the lowest point of initial calibration (ICAL), which should be at least 3 times the applicable MDL.

11.0 CALCULATIONS

11.1 Calculation of calibration factors (CF):

Tabulate the peak area against concentration for each compound and each standard.

Calculate calibration factors (CF) for each compound as shown below:

$$CF = (A_x) / (C_x)$$

where:

Ax = Peak area for the compound being measured

Cx = Concentration of the compound (ng/ml) in the standard

11.2 Calculation of mean calibration factors (mean CF or CF_m):

For each compound, calculate the mean or average calibration factor (mean CF = CF_m) using the five or six CF values calculated for the 5 or 6 standard solutions as shown below:

$$\text{mean CF} = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

Where CF_i are the calibration factors for standards 1 to 5. Calculate the average as shown above using appropriate number of CF_i, if the number of standards (n) analyzed exceeds 5 for specific compounds. Use Excel spreadsheets, laboratory information system (LIMS) or the instrument software to determine mean, standard deviation (See Section 11.3), RSD, linear regression equations, correlation coefficients, etc. Although non-linear equations for the

ICAL may be employed in selected instances, its use for routine sample analysis is discouraged.

11.3 Calculation of standard deviations and %RSDs:
Precision for the initial calibration (ICAL) may be expressed as the standard deviation (SD), percent relative standard deviation (RSD), or coefficient of variation (CV). Calculate RSD for the ICAL substituting CF_i for X_i and CF_m for mean x (X_m). The number "n" is the number of standards used to calculate the mean.

$$RSD = CV = \frac{SD * 100}{X_m}$$

$$SD = \left\{\frac{\Sigma(X_i - X_m)^2}{(n-1)}\right\}^{1/2}$$

$$X_m = \frac{\Sigma(X_i)}{n}$$

11.4 Calculation of %D for calibration verification:
Calculate the percent drift or the percent difference (%D) between the observed calibration factor for the verification solution (CF_v) and the mean calibration factor from the ICAL (CF_m) as shown below:

$$\%D = ((CF_v - CF_m) * 100 / (CF_m))$$

Percent drift is calculated when linear regression is used for ICAL. See Section 7.7.1 of Method 8000B for the calculation of %Drift.

11.5 Percent Recovery (%R) for LCS/LCSD and, MS/MSD samples:
Percent recovery (%R) is calculated as follows.

$$\%R = \frac{(x_s - x_u)}{K} x\,100$$

where:

X_s = measured sample concentration of the spiked sample

X_u = measured sample concentration of the unspiked sample

K = known concentration of the spike in the spiked sample

When calculating %R for LCS or other reference materials, X_u could be set at zero. The same procedure should be used for calculating surrogate recoveries.

11.6 Relative percent difference (RPD) for duplicates:
Precision for duplicate analysis is estimated from the relative percent difference (RPD) of the concentrations (not the recoveries) measured for MS/MSD pairs, LCS/LCSD pairs or for duplicate analyses of unspiked samples. Calculate RPD as shown below:

$$RPD = \frac{|(C_1 - C_2)| * 100}{(C_1 + C_2) * (0.5)}$$

where:
C_1 = Measured concentration of the first aliquot
C_2 = Measured concentration of the second aliquot

Calculate sample concentrations for water and soil samples as described in Sections 11.7 and 11.8 respectively. This equation may also be used to calculate RPD between sample results obtained from dual chromatography columns.

11.7 Calculation of sample concentrations in water samples:
For water samples the calculation is as follows:

$$\text{Concentration } (\mu g/L) = \frac{(Ax * V_e * D)}{(CF_m * V_s)}$$

where:
A_X = Peak area of the compound being measured.
CF_m = Mean calibration factor ($(ng/ml)^{-1}$) for compound being measured
V_e = Volume (ml) of the final extract (default = 5-mL)
V_s = Volume of water (ml) extracted (default = 1000-ml)
D = Dilution factor for the analysis

11.8 Calculation of sample concentrations in solid (soil, sediment or waste) samples:
For solid samples the calculation is as follows:

$$\text{Concentration } (\mu g/kg) = \frac{(Ax * V_e * D)}{(CF_m * W_s)}$$

where:
A_X = Peak area of the compound being measured.
CF_m = Mean calibration factor ($(ng/ml)^{-1}$) for compound being measured
V_e = Volume (ml) of the final extract (default = 5-mL)
W_s = Weight of solid sample (g) extracted (default = 30-g)
D = Dilution factor for the analysis

Solid samples for this analysis are air-dried prior to analysis. Therefore, corrections are not made for %solids in the sample.

12.0 DATA VERIFICATION AND VALIDATION - This section will provide guidance when evaluating and validating data generated using this SOP. Using this SOP will involve implementing the referenced EPA method at the laboratory. Method implementation involves the following: chromatographic separation of the target analytes on two HPLC columns, determination of retention time windows for each compound, determination of method detection limits for each sample matrix and/or preparation method, spiked sample analysis, initial demonstration of acceptable recoveries, etc. Method implementation procedures are described on the AML SOP on MDL. This SOP provides procedures to assess if data produced using this SOP are valid and legally defensible. Document review and comments on a checklist (see Appendix 6 for details). Verify that sample preparation logs are complete and the instrument run logs are available for review (see Appendix 13 for more details). Anomalies and exceptions should be documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on the checklists. USACE projects may have special requirements (Section 12.1) that will require extra attention. A simple, universal procedure for evaluating and choosing appropriate corrective action doe not exist to deal with situations when one or more QC parameters are outside the acceptance window. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints.

12.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) projects may have additional requirements that are related to USACE laboratory validation (CX), USACE District requirements, government contract requirements, specific USACE project requirements or USACE Architect/Engineer (A/E) requirements. USACE-CX requirements (Summary of Method Quality Objectives for Method 8330) are included in the cited references (see Appendix 8 for details).

12.1.1 The USACE requirements shown (see Appendix 8 for details) elsewhere in this SOP are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may or may not be attainable for routine sample analysis. Since USACE MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

12.1.2 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality objectives

and/or other QC requirements. Exceptions and data qualifications necessary should be documented on the checklists and eventually on case narratives.

12.1.3 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist, perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete. For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete. For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director will review the checklists (initial/date as necessary), prepare case narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

12.1.4 The USACE blank criteria (see Appendix 8 for details) have been modified by USACE auditor(s) as one half of the applicable MQL (personal communications, USACE auditor, 1999).

12.2 HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

12.2.1 CHECK- The holding times for extraction of water samples is 7 days from sampling. Soil samples should be extracted within 14-days of collection. TCLP leachates should be prepared within 14-days of soil or waste sample collection. TCLP leachates should be extracted for target compounds within 7-days of leachate preparation. Sample extracts should be analyzed within 40-days of extraction.

12.2.2 CHECK - Actual holding times are determined by comparing the sampling date on the chain of custody with the date of extraction or leachate (TCLP) preparation. Actual holding times for analysis are determined by comparing the extraction date to the injection date.

12.2.3 ACTION – If holding times have been exceeded, contact the client. All positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or invalid (I).

12.3 INITIAL CALIBRATION – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the instrument is capable of producing data within a range that is linear (see Appendix 2 for details).

12.3.1 CHECK – (a) Check to see if any compounds are outside the QC criteria of 20% or less for %RSD. (b) If one or more compounds are outside the 20% criteria, check to see that the mean %RSD for all the compounds is less than 20%. (c) Check to see that %RSD is <40% for all compounds (USACE criteria). (d) If %RSD is >20% but <40%, check to see if linear regression was used for the affected compounds. (e) If linear regression was used, check to see

that correlation coefficient for linear regression is 0.990 or higher for each affected compound.

12.3.2 ACTION – If the checks (a) to (e) pass, the ICAL is acceptable. If check (c) or (e) fails, generate a new ICAL for USACE projects. Data qualification may be used for non-USACE projects only. Document observations on the checklist (see Appendix 6 for details).

12.4 CALIBRATION VERIFICATION – It consists of two parts: initial calibration verification (ICV) described in Sections 10.2.4 and 10.2.5; and continuing calibration verification, which is described in Sections 10.2.6 and 10.2.7. ICV is run immediately after the initial calibration to find any potential systematic errors on calibration standards. CCVs are used to check that the instrument is still operating under the linear range of the initial calibration and it has remained stable by checking before, during, and after sample analysis (see Appendix 3 for details).

12.4.1 CHECK – Calculate %D for each calibration verification (CALV), which includes initial calibration verification (ICV) and continuing calibration verifications (CCV). (a) Check to see if the %D for each target compound is less than 15%. (b) If one or more compounds have %D >15%, check to see that the %D is <15% for the contaminants of concern (COC) compounds. (c) Check to see that the mean %D for all the target compounds is <15%. (d) Check to see that %D for each target compound is <30% (USACE criteria).

12.4.2 ACTION - If the %D is outside the QC limits for any compound in the ICV, check the instrument, ICAL standards and the ICV standard for anomalies. Replace the ICV standard first if the problem cannot be traced to the ICAL standards. Replace ICAL standards, if the problem is not corrected by replacing the ICV standard. Generate a new ICAL.

12.4.3 ACTION – The CCVs are acceptable if (b) and (c) pass the check. The CCVs are acceptable for USACE projects if it also passes (d). The CCVs are acceptable for any project if they pass check (a). If the affected compound is a contaminant of concern (COC), correct the problem and re-analyze affected samples. Treat compounds that are positive as COC. If the affected compound is not one of the COC compounds, and the CCV is acceptable, data qualification may be an option. If data qualification is not an option, repeat CALV and reanalyze affected samples. A new ICAL and an ICV will be necessary if acceptable CCVs cannot be obtained. Document observations on a checklist (see Appendix 6 for details).

12.4.4 ACTION - Data qualifications are performed by the end-user and information for data qualification is provided by the laboratory. Report detects and non-detects without qualifiers and document QC outliers in the case narrative. Results for compounds not meeting criteria may be qualified as estimated (J for positives and UJ for non-detects) by the data user (client).

12.5 METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Sporadic cases of contamination may be difficult to control. However, daily contamination would not be acceptable. The concentration of target analytes should not exceed QC criteria (e.g. one half of the applicable MQL for USACE projects, See Section 12.1.4). If they do, steps

shall be taken to find/reduce/eliminate the source of this contamination in the method blank with the exception of common laboratory contaminants (None for this analysis). Additional project-specific corrective actions may be necessary. Sample data qualification to alert blank contamination is performed by the laboratory. Sample data qualification based on all the blank results (laboratory MB, equipment blank, and other field blanks) is performed by the user (client). The processing of blanks helps eliminate reporting of false positives in project reports. To be acceptable, blank results should not exceed blank criteria. Since uniform criteria for an acceptable MB is unavailable, the laboratory will use the USACE criteria (<0.5*MQL) for all target compounds. See Tables 1 and 2 for a list of MQLs.

12.5.1 CHECK - Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples.

12.5.2 ACTION – Report all positive sample results with a "B" code, if contaminants are found in the method blank at levels greater than MDL or method reporting limit, whichever is used for reporting sample data for the project.

12.5.3 ACTION – If a compound is detected only in the blank and not in any of the associated samples, then no action is taken. Discuss the situation in the case narrative.

12.5.4 ACTION – Any compound detected in the sample which is also detected in associated method blank at levels greater than one half the MQL correct the problem by re-extracting associated samples unless other client criteria are applicable. Exceptions may include quick turn around projects. Contact client.

12.5.5 ACTION – If the sample amount is more than 10-times the blank amount, re-extraction and re-analysis of the affected sample may not be necessary if the contamination of the MB is associated with the processing of grossly contaminated samples.

12.5.6 ACTION - If the sample amounts are less than 10-times the blank level, reanalyze the method blank, and any samples containing the same contaminant. If the contamination remains, the contaminated samples of the batch should be re-prepared and reanalyzed with a new method blank and batch specific QC samples.

12.6 SURROGATE RECOVERIES – Surrogates or system monitoring compounds are different structurally, but similar chemically to classes of target compounds. These surrogate compounds are added just prior to extraction of the samples and the percent recoveries of the surrogates are calculated and evaluated. A surrogate is evaluated by comparing its recovery (%R) in each sample to the laboratory QC limits, project-specific DQOs, client specifications, or other criteria (e.g. EPA limits). Document surrogate recoveries on Form-2 equivalents (See Appendix 11 for details).

12.6.1 Surrogate failures in method blanks or laboratory control samples are indicative of a general method failure and should be thoroughly investigated. Samples that appear to be heavily contaminated either with target or non-target analytes may require method modification (such as the medium level method instead of low level method) even for an initial analysis. Such requests may either originate from the analyst or the client project chemist. Other consequences such as higher detection limits for the medium level analysis,

additional cost for low-level re-analysis, if medium level analysis is unacceptable, etc. should also be considered in this instance. Surrogate recoveries that fail to meet appropriate acceptance criteria would indicate that a potential matrix effect is present. If significant non-target interference occurs, corrective action shall include implementing additional cleanup procedures, and reanalyses. If this does not reduce the interference, discuss the impact on the data within the case narrative. For USACE projects, tighter limits may apply to interference-free samples such as MB, LCS and LCSD.

12.6.2 CHECK - Verify that the surrogate (1-chloro-3-nitrobenzene) recoveries are within QC limits for the environmental samples including MS and MSD (50% to 150%) and interference-free samples including MB, LCS and LCSD (60% to 140% for water and 50% to 150% for soil). Flag outliers with an asterisk (*).

12.6.3 ACTION – When surrogate recoveries are outside control limits, the sample must be re-analyzed. Reanalysis may be performed under dilution if original analysis indicates high levels of target and/or non-target analytes. The SW-846 document does not contain any recommendation for cleanup procedures for Method 8330 analytes. If surrogate recoveries are still outside the limits after dilution, report both sets of data and describe the problem in the case narrative.

12.6.4 ACTION - If method modifications are needed, contact the client for authorization. The authorization may involve request for additional payment before reanalysis may begin.

12.7 LABORATORY CONTROL SAMPLE (LCS) – The analysis of LCS samples is used to assess method performance in the laboratory in the absence of matrix effects. The LCS is normally from a second source standard. In response to the USACE initiatives (See USACE Shell document, EM-200-1-3, Appendix I) the laboratory will be standardizing on LCS samples based on the same source as ICAL standards. The LCS is evaluated by comparing the percent recovery for all of the target analytes to the laboratory QC limits, which are identical to the USACE method quality objectives (see Appendix 8 for details). Report LCS and LCSD concentrations in Form-1 equivalents and %R in Form-3 equivalents (see Appendix 10 for details). Other evaluation procedures may include comparison with those determined by project-specific DQOs (See project QAPP, if available), laboratory statistical control limits, or USEPA guidance. Initially, the effect of QC failure on the samples should be evaluated. Regardless of this assessment, steps shall be taken to find the source of the problem and correct it.

12.7.1 CHECK - Compare the %R results for each compound in LCS with laboratory QC limits (70% to 130% for soil and water) to see if all the results are within the acceptable range. Flag outliers with an asterisk (*). NOTE: AML laboratory limits have been modified from USACE limits (60% to 120%) since average %R at AML are closer to 100% rather than 90%.

12.7.2 ACTION – A general rule is to have at least 90% of the target analytes to have %R within control limits. The supervisor should be consulted if this is not achievable. For USACE projects, acceptance criteria may allow for sporadic marginal failure criteria (40% to 150%) for the %R of a few target analytes

(0,1, and 2) when multiple analytes (<5, 5-15, and 16-30 respectively) are reported for a project. Typically, the LCS would be reanalyzed for the failed analytes only. If the second analysis fails, then the LCS, method blank, and all associated samples of the batch would be re-prepared and reanalyzed for the failed analytes only. If sufficient sample is not available for re-preparation and reanalysis, or if the corrective action is ineffective, the sample results reported within that batch shall be flagged accordingly (*), and a discussion of the impact included within the case narrative. The case narrative should discuss the corrective action taken and any other information. For non-USACE projects, no action may need to be taken on LCS and/or LCSD recovery data alone either to reanalyze samples and/or to qualify associated samples. If significant deviations are noticed, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements, which may be compound specific. Check with the project manager about such requirements. Professional judgment may be used by the user (client) in evaluating sample data qualification options in conjunction with other QC data for the project.

12.8 MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERIES – Matrix spike recovery data are used to evaluate the accuracy of the analytical method on project samples. The evaluation is performed by comparing the %R for target analytes to the laboratory statistical control limits, laboratory QC limits, other recovery windows established within project documents, client specification, etc. Document MS results in Form-1 equivalents and recoveries in Form-3 equivalents (See Appendix 9 for details).

12.8.1 MS data evaluation is more complex than method blank or LCS data evaluation since they are complicated by matrix effects in addition to sample preparation and analysis errors. The heterogeneity of grab soil samples, and potential non-representativeness of sequentially collected water samples, etc. further complicate the evaluation since it is assumed that the native concentrations are constant in triplicate analyses. In addition, concentrations of the target analytes in the sample can also far exceed spike amounts. If the native concentration of target analytes in the sample chosen for spiking is high relative to the spiking concentration, the differences in the native concentration between the unspiked sample and the spiked samples may also become very significant.

12.8.2 In addition to their reluctance to pay for MS and/or MSD analyses, most clients may not know what samples to collect for MS analysis, may not collect sufficient samples, may not have any knowledge of the native analyte concentrations in the chosen sample, and may not have a knowledge of MS spiking levels that are appropriate either for the sample or their project. For procedures with multiple target analytes, spiking levels may be inappropriate for one or more analytes, if samples that contain target analytes are chosen either in the field or by the laboratory. If no project samples are collected for MS analysis, samples from another project used for MS analysis may not be representative of the project samples. When project samples (e.g. surface soil) are chosen, their MS recovery characteristics may be different from those of other samples (e.g. sub-surface soil) in the same batch.

12.8.3 Laboratory project managers should discuss MS requirements with client personnel to ensure that their requirements match with those of their clients

and they are achievable on a routine basis by commercial laboratories. Three separate aliquots of samples collected for unspiked and spiked (MS and MSD) sample analyses may be combined, carefully homogenized and subsamples used for the triplicate analysis..

12.8.4 The laboratory QC limits for MS/MSD recoveries are shown in Tables 1 and 2. The laboratory QC limits have been adopted from USACE method quality objectives document (see Appendix 8 for details). Refer to project data quality objective data (included in project QAPP, when available) for appropriate corrective action.

12.8.5 CHECK - Compare %R results for each compound with the laboratory limits for MS/MSD recoveries (50% to 140% for soil and water). Flag outliers with an asterisk (*). For USACE projects, acceptance criteria may allow for sporadic marginal failure criteria (40% to 150%) for the %R of a few target analytes (0,1, and 2) when multiple analytes (<5, 5-15, and 16-30 respectively) are reported for a project. Perform manual calculation for %R using raw data and extraction sheet information to check outliers.

12.8.6 ACTION – The results of the MS are evaluated, in conjunction with other QC information, to determine the effect of the matrix on the bias of the analysis. A general rule is to have 95% of the target analytes to have %R within QC limits. The supervisor should be consulted if this is not achievable. For USACE projects, acceptance criteria may allow for sporadic marginal failure criteria (40% to 150%) for the %R of a few target analytes (0,1, and 2) when multiple analytes (<5, 5-15, and 16-30 respectively) are reported for a project. Regardless, steps should be taken to find the cause of failure and corrective action to remedy it. MSs that fail to meet the appropriate acceptance criteria may indicate that a potential matrix effect is present. A review of the MSD result, if available, may confirm the matrix effect, if it is in the same direction and same order of magnitude.

12.8.7 ACTION - If the sample chosen for MS/MSD is inappropriate, notify the client. When additional payments are authorized for re-analysis, reextract and/or reanalyze the MS/MSD samples after employing cleanup procedures (if applicable), dilution techniques to minimize matrix interference, or using other project samples that appear to be clean, when sufficient quantities are available. Re-spike the sample at a higher level (e.g., at approximately two to four times the sample concentration), then reanalyze the sample based on project-specific requirements. It is the responsibility of the client at all times to select appropriate samples for MS analysis, make additional payments for additional analyses, and to offer necessary guidance as needed to meet their data quality objectives.

12.8.8 If the matrix effect cannot be resolved, discuss the situation in the case narrative. No action is taken by the laboratory on MS/MSD recoveries data alone to qualify associated samples. The user (client) may use Professional judgment in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

12.9 DUPLICATES - Duplicates include sample or matrix duplicates (MD), matrix spike duplicates (MS/MSD), and LCS duplicates. The precision is evaluated by comparing the observed RPD between the two sets of results (concentrations and not %R) with

laboratory QC limits (50% for soil and water) to see if all the results are within the acceptable range. See Appendices 9 and 10 for guidance on documenting RPDs for LCS/LCSD, MS/MSD and M/MD pairs in QC summary report forms. Flag outliers with an asterisk (*). For USACE projects, acceptance criteria may allow for sporadic marginal failure (SMF) criteria (60%) for a few target analytes (0,1, and 2 respectively) when multiple analytes (<5, 5-15, and 16-30 respectively) are reported for a project. These criteria should only be applied to concentrations of target analytes that are above each analyte's MQL. MDs or MSDs that fail to meet acceptance criteria may indicate either sample heterogeneity or the presence of potential matrix effects.

12.9.1 CHECK - Compare the RPD results for each compound in the duplicate set (LCS/LCSD, MS/MSD or M/MD) with laboratory QC limits (50% for soil and water and 60% for SMF analytes) to see if all the results are within the acceptable range. Flag outliers with an asterisk (*).

12.9.2 ACTION - LCS duplicates are performed by the laboratory to monitor precision on a routine basis. Reextractions and/or reanalyses are not required if RPDs for LCS/LCSD sets are outside QC limits. A general rule is to have at least 90% of the target analytes to have %R within control limits for MS/MSD and/or M/MD sets as appropriate. The supervisor should be consulted if this is not achievable. For USACE projects, acceptance criteria may allow for sporadic marginal failure (SMF) criteria (40% to 150%) for RPD of a few target analytes (0,1, and 2 respectively) when multiple analytes (<5, 5-15, and 16-30 respectively) are reported for a project.

12.9.3 ACTION - The RPDs for MS/MSD and M/MD sets have problems that are similar to that described for MS/MSD %R data. See Section 12.7 for additional guidance. Authorization by client (to cover costs for reanalysis) may be necessary. Check with the project manager about project specific requirements. Professional judgment may be used by the user (client) in evaluating sample data qualification options in conjunction with other QC data for the project.

12.10 TARGET COMPOUND IDENTIFICATION AND REPORTING – In the absence of project-specific criteria, minimize the possibility of incorrect identification (or false positives) through confirmation on a second dissimilar chromatographic column for single component target analytes. After the target analyte has been identified, compare the primary and confirmatory results for agreement according to method-prescribed criteria. Analytical results would normally be reported from the primary column unless interferences were noted. Report sample data using Form-1 (see Appendix 7 for details).

12.10.1 CHECK - Positive compounds must be within +/- 0.06 relative retention time (RRT) units of the standard RRT on both columns. The RPD between quantitative results from the two columns for positively identified compounds should be <40%.

12.10.2 ACTION – Report data from the primary column using data from the confirmation column for RPD determination only unless interferences are noted (See Section 10.3 for additional guidance). Report target compounds that are present at levels greater than either the MDL (see Appendix 12 for details on MDL studies) or the RL (as appropriate for the project) using "J" qualifiers if necessary, if the levels are less than MQL. See Section 10.3.10 for a discussion of MDL, RL, and MQL. Compounds that were also detected in the

associated MB should also be qualified with a "B" code. If the concentration of any of the target compounds exceeds the upper limit of calibration (ICAL), flag the data for affected compounds with an estimated or "E" flag. The extracts may need to be diluted and reanalyzed until the concentrations are within the calibration limits for normal turnaround analyses. For rapid turnaround analyses and for compounds that are not contaminants of concern for specific projects, sample results may be reported with "E" qualifiers. Use additional QC flags as described in the following section.

12.10.3 Data qualifiers shall be added by the analyst and/or the laboratory during data generation, verification, review, or reporting. The following data qualifiers are suggested for normal use:

12.10.4 U - Non-detect when analyte concentration is below MDL or MRL as appropriate

12.10.5 J - Estimated concentration when analyte concentration falls below MQL

12.10.6 J - Estimated concentration when the RPD between the analyte concentrations from the primary and confirmation columns is greater than 40%

12.10.7 B - Blank contamination when any associated blanks are above the MDL or MRL

12.10.8 E- Estimated concentration when analyte concentrations exceed the upper limit of initial calibration

12.10.9 D - Data reported are from method of standard addition (not applicable to this procedure).

12.10.10 I - Invalid results when target and/or non-target interference does not allow a valid result to be reported for a target analyte

12.10.11 R - Rejected due to problems that cannot be resolved by the laboratory within schedule to report meaningful data for the analyte. This flag can only be used by the Level 3 Reviewer.

12.10.12 The QC flags described above may be used to identify any suspected bias in the data, either low or high, and whether the estimation is related to the suspected identification (qualitative) or whether the value reported is an approximation (quantitative). The project manager or appropriate technical personnel should be notified as soon as possible to discuss possible corrective actions for data requiring qualifications. Additional data flagging may be performed during external data review or validation by the client based on project specific requirements, EPA guidance, etc.

13.0 REFERENCES

13.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8310, Polynuclear Aromatic Hydrocarbons, September 1986.

13.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.

13.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

13.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3500B, Organic Extraction and Sample Preparation, December 1996.

13.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3500C, Organic Extraction and Sample Preparation, Draft Update IVB.

13.6 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3535, Solid Phase Extraction, December 1996.

13.7 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3535A, Solid-Phase Extraction, Draft Update IVA and IVB.

13.8 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3510C, Separatory Funnel Liquid-Liquid Extraction, December 1996.

13.9 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3520C, Continuous Liquid-Liquid Extraction, December 1996.

13.10 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3540C, Soxhlet Extraction, December 1996.

13.11 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3541, Automated Soxhlet Extraction, December 1996.

13.12 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3550B, Ultrasonic Extraction, December 1996.

13.13 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3600C, Cleanup, December 1996.

13.14 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3630C, Silica Gel Cleanup, December 1996.

13.15 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3640A, Gel-Permeation Cleanup, December 1996.

13.16 EM 200-1-1, Validation of Analytical Chemistry Laboratories, US Army Corps of Engineers. July 1, 1994.

13.17 EM 200-1-2, Technical Project Planning (TPP) Process, US Army Corps of Engineers, August 31, 1998.

13.18 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, Appendix I, US Army Corps of Engineers.

13.19 EM 200-1-6, Chemical Quality Assurance, US Army Corps of Engineers, October 10, 1997.

13.20 ER 1110-1-263, Chemical Data Quality Management for Hazardous, Toxic, Radioactive Waste Remedial Activities, US Army Corps of Engineers, April 30, 1998.

13.21 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

13.22 AML Standard Operating Procedures, Q-Water-RC.

13.23 AML Standard Operating Procedures, Q-WasteMgmt-RD.
13.24 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
13.25 AML Standard Operating Procedures, Q-Controlcharts-RC.

Table 1
Aqueous MDL, MQL and QC Limits for LCS and MS

Polynuclear Aromatic Hydrocarbons (PAHs) Analysis by HPLC

Analytical Management Laboratories

Matrix: Water	Preparation Method			3510C										
	Analytical Method			8310										
	Spike	Mean	S.D.	**MDL**			**MQL**			**DoD QSM LCS & MS Control Limits**		**DoD QSM LCS & MS ME Limits**		**RPD**
Compound	Amount	ug/L	ug/L	**ug/L**			**ug/L**			Lower	Upper	Lower	Upper	Limit
	ug/L	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%
Naphthalene	4.00	3.99	0.070	**0.22**			1.0			35	105	20	115	50
Acenaphthylene	5.00	5.02	0.085	**0.27**			1.0			35	115	20	125	50
Acenaphthene	0.70	0.70	0.026	**0.08**			0.5			35	105	25	115	50
Fluorene	0.60	0.61	0.057	**0.18**			1.0			35	105	25	115	50
Phenanthrene	0.20	0.16	0.024	**0.08**			0.5			40	120	25	135	50
Anthracene	0.15	0.13	0.024	**0.08**			0.5			40	110	30	125	50
Fluoranthene	0.50	0.50	0.015	**0.05**			0.5			50	115	35	125	50
Pyrene	1.00	0.97	0.012	**0.04**			0.5			50	110	45	115	50
Benzo(a) anthracne	0.50	0.49	0.118	**0.37**			1.0			50	110	40	125	50
Chrysene	0.50	0.49	0.019	**0.06**			0.5			50	115	40	125	50
Benzo(b)fluoranthene	0.20	0.22	0.002	**0.01**			0.5			50	110	40	125	50
Benzo(k)fluoranthene	0.20	0.21	0.004	**0.01**			0.5			50	110	40	120	50
Benzo(a) pyrene	0.50	0.49	0.018	**0.06**			0.5			45	115	35	125	50
Dibenz(a,h)anthracene	0.50	0.68	0.205	**0.64**			2.0			20	110	10	125	50
Benzo(g,h,i)perylene	0.40	0.34	0.039	**0.12**			0.5			35	120	20	135	50
Indeneo(1,2,3-cd)pyrene	0.25	0.26	0.024	**0.07**			0.5			45	110	35	125	50

Control and marginal exceedance (ME) limits for LCS recoveries (%R) are from DoD QSM, Final Version 2, June 2002.

DoD QSM Control and marginal exceedance (ME) limits for LCS recoveries (%R) were also applied to MS recoveries.
Limits for RPD are from the USACE EM200-1-3, Appendix I, I Feb 2001.

The following criteria will apply for marginal exceedances:

Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).

NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.

Table 2
Soil/Sediment MDL, MQL and QC Limits for LCS and MS

Polynuclear Aromatic Hydrocarbons (PAHs) Analysis by HPLC														
Analytical Management Laboratories														
Matrix: Soil	Preparation Method			3550C										
	Analytical Method			8310										
	Spike	Mean	S.D.	**MDL**			**MQL**			**DoD QSM LCS & MS Control Limits**		**DoD QSM LCS & MS ME Limits**		**RPD**
Compound	Amount	ug/kg	ug/kg	**ug/kg**			**Ug/kg**			Lower	Upper	Lower	Upper	Limit
	ug/kg	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%
Naphthalene	200	136	2.21	**6.95**			33.3			50	110	40	120	60
Acenaphthylene	250	167	3.60	**11.3**			33.3			35	115	20	125	60
Acenaphthene	35	23.0	1.05	**3.30**			33.3			35	110	20	125	60
Fluorene	30	26.2	3.93	**12.3**			33.3			45	105	35	115	60
Phenanthrene	10	6.80	0.98	**3.07**			33.3			55	125	45	135	60
Anthracene	7.5	15.4	4.41	**13.9**			33.3			45	125	35	140	60
Fluoranthene	25	22.0	7.59	**23.8**			66.7			40	135	25	150	60
Pyrene	50	29.7	4.67	**14.7**			33.3			50	115	40	125	60
Benzo(a) anthracne	25	24.6	0.50	**1.56**			33.3			50	105	40	115	60
Chrysene	25	20.3	3.46	**10.9**			33.3			55	120	45	130	60
Benzo(b)fluoranthene	10	7.29	0.22	**0.70**			3.33			55	120	45	130	60
Benzo(k)fluoranthene	10	8.27	3.06	**9.62**			33.3			50	120	35	135	60
Benzo(a) pyrene	25	16.6	1.23	**3.85**			33.3			40	135	25	150	60
Dibenz(a,h)anthracene	25	18.8	5.66	**17.8**			66.7			45	115	35	125	60
Benzo(g,h,i)perylene	20	11.2	3.30	**10.4**			33.3			55	115	45	125	60
Indeneo(1,2,3-cd)pyrene	12.5	8.35	0.13	**0.40**			33.3			55	135	45	145	60
Control and marginal exceedance (ME) limits for LCS recoveries (%R) are from DoD QSM, Final Version 2, June 2002.														
DoD QSM Control and marginal exceedance (ME) limits for LCS recoveries (%R) were also applied to MS recoveries. Limits for RPD are from the USACE EM200-1-3, Appendix I, I Feb 2001.														
The following criteria will apply for marginal exceedances:														
Number of allowed ME analytes per (total analytes): 0 (<11), 1 (11-30), 2 (31-50), 3 (51-70), 4 (71-90), and 5 (>90).														
NOTE: MDLs and MQLs are dynamic parameters. See the SOP appendices for recent MDL and MQL data.														

Table 3
List of Appendices

Appendix Number	Appendix Topic
1	Stock Standards, Concentrations and Typical Expiry Dates
2	Initial Calibration (ICAL) Summary (Form-6 equivalents) Instrument Printouts and Excel Spreadsheets
3	Calibration Verification (ICV and CCV) Summary (Form-7 equivalents) Instrument Printouts and Excel Spreadsheets
4	Standard Chromatograms for CCV (C-18 PAH Column) Instrument Printouts
5	Standard Chromatograms for CCV (Other PAH Columns) Instrument Printouts
6	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
7	Sample Reports (Form-1 equivalents)
8	USACE Method Quality Objectives for Method 8310 AFCEE MQOs for Method 8310 DoD QSM (Final Revision 2) Method Quality Objectives
9	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
10	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
11	Surrogate Recovery Reports (Form-2 equivalents)
12	Method Detection Limit (MDL) Studies Summary
13	QC Association Forms (Form-4 equivalents) Sample Preparation Logsheets Instrument Run Log

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. I-Corrosivity-SoilpH-RC

ELECTROMETRIC MEASUREMENT of pH IN SOIL AND WASTE (EPA SW846-9045D)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

ELECTROMETRIC MEASUREMENT of pH IN SOIL AND WASTE (EPA SW846-9045D)

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) is based on EPA SW-846 Method 9045C. This SOP is used to measure the pH in soils, sludges, non-aqueous liquids and other wastes in which the aqueous phase constitutes less than 20% of the total volume of the sample.

2.0 SUMMARY OF METHOD

2.1 The sample is mixed with reagent water, and the pH of the resulting aqueous solution is measured.

3.0 INTERFERENCES

3.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode. Strong acid solutions, with a true pH of <1, may give incorrectly high pH measurements.

3.2 Temperature fluctuations will cause measurement errors.

3.3 Errors will occur when the electrodes become coated. If an electrode becomes coated with an oily material that will not rinse free, the electrode can (1) be cleaned with an ultrasonic bath, or (2) be washed with detergent, rinsed several times with water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with water, or (3) be cleaned per the manufacturer's instructions.

4.0 APPARATUS AND MATERIALS

4.1 pH Meter with means for temperature compensation.

4.2 Glass Electrode.

4.3 Reference electrode: A silver-silver chloride or other reference electrode of constant potential may be used.

> NOTE: Combination electrodes incorporating both measuring and referenced functions are convenient to use and are available with solid, gel-type filling materials that require minimal maintenance.

4.4 Beaker: 50-mL.

4.5 Thermometer and/or temperature sensor for automatic compensation.

4.6 Analytical balance: capable of weighing 0.1 g.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Reagent water. All references to water in this method refer to reagent water, as defined in Chapter One.

5.3 Primary standard buffer salts are available from the National Institute of Standards and Technology (NIST) and should be used in situations where extreme accuracy is necessary. Preparation of reference solutions from these salts requires some special precautions and handling, such as low-conductivity dilution water, drying ovens, and carbon-dioxide-free purge gas. These solutions should be replaced at least once each month.

5.4 Secondary standard buffers may be prepared from NIST salts or purchased as solutions from commercial vendors. These commercially available solutions, which have been validated by comparison with NIST standards, are recommended for routine use.

6.0 SAMPLE PRESERVATION AND HANDLING

6.1 All samples must be collected using a sampling plan that addresses the considerations discussed in Chapter Nine of the SW-846 manual.

6.2 Samples should be analyzed as soon as possible.

7.0 PROCEDURE

7.1 Calibration:

7.1.1 Because of the wide variety of pH meters and accessories, detailed operating procedures cannot be incorporated into this method. Each analyst must be acquainted with the operation of each system and familiar with all instrument functions. Special attention to care of the electrodes is recommended.

7.1.2 Each instrument/electrode system must be calibrated at a minimum of two points that bracket the expected pH of the samples and are

approximately three pH units or more apart. Repeat adjustments on successive portions of the two buffer solutions until readings are within 0.05 pH units of the buffer solution value. If an accurate pH reading based on the conventional pH scale [0 to 14 at 25°C] is required, the analyst should control sample temperature at 25±1°C when sample pH approaches the alkaline end of the scale (e.g., a pH of 11 or above).

7.2 Sample preparation and pH measurement of soils:

7.2.1 To 20 g of soil in a 50-mL beaker, add 20 mL of reagent water, cover, and continuously stir the suspension for 5 minutes. . Additional dilutions are allowed if working with hygroscopic soils and salts or other problematic matrices.

7.2.2 Let the soil suspension stand for about 1 hour to allow most of the suspended clay to settle out from the suspension or filter or centrifuge off the aqueous phase for pH measurement.

7.2.3 Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant solution to establish a good electrical contact through the ground-glass joint or the fiber-capillary hole. Insert the electrodes into the sample solution in this manner. For combination electrodes, immerse just below the suspension.

7.2.4 If the sample temperature differs by more than 2°C from the buffer solution, the measured pH values must be corrected.

7.2.5 Report the results as "soil pH measured in water at __°C" where "_°C" is the temperature at which the test was conducted.

7.3 Sample preparation and pH measurement of waste materials:

7.3.1 To 20 g of waste sample in a 50-mL beaker, add 20 mL of reagent water, cover, and continuously stir the suspension for 5 minutes. . Additional dilutions are allowed if working with hygroscopic wastes and salts or other problematic matrices.

7.3.2 Let the waste suspension stand for about 15 minutes to allow most of the suspended waste to settle out from the suspension or filter or centrifuge off aqueous phase for pH measurement.

> NOTE: If the waste is hygroscopic and absorbs all the reagent water, begin the experiment again using 20 g of waste and 40 mL of reagent water.

NOTE: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous phase. The electrode may need to be cleaned (Step 3.3) if it becomes coated with an oily material.

7.3.3 Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant to establish good electrical contact through the ground-glass joint or the fiber-capillary hole. Insert the electrode into the sample solution in this manner. For combination electrodes, immerse just below the suspension.

7.3.4 If the sample temperature differs by more than 2°C from the buffer solution, the measured pH values must be corrected.

7.3.5 Report the results as "waste pH measured in water at _°C" where "_°C" is the temperature at which the test was conducted.

8.0 QUALITY CONTROL

8.1 Refer to AML quality assurance plan.
8.2 Electrodes must be thoroughly rinsed between samples.

9.0 REFERENCES

9.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 6, Properties, December 1996.
9.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 7, section 7.2.2(1)(a), Characteristics Introduction and Regulatory Definitions, December 1996.
9.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9045C, Soil and Waste pH, January 1995.
9.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9045D, Soil and Waste pH, Update IIIB.
9.5 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.
9.6 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.
9.7 AML Standard Operating Procedures, Q-Water-RC.
9.8 AML Standard Operating Procedures, Q-WasteMgmt-RD.
9.9 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
9.10 AML Standard Operating Procedures, Q-Controlcharts-RC.

STANDARD OPERATING PROCEDURES

No. W-Corrosivity-Steel-RC

CORROSIVITY TO STEEL DETERMINATION (SW-846 Method 1110A)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

CORROSIVITY TO STEEL DETERMINATION (SW-846 Method 1110A)

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) is based on SW-846 Method 1110 to measure the corrosivity toward steel of both aqueous and nonaqueous liquid wastes. This SOP is not as commonly used as AML SOP aml9045C, which determines corrosivity through pH measurements.

2.0 SUMMARY OF METHOD

This test exposes coupons of SAE Type 1020 steel to the liquid waste to be evaluated and, by measuring the degree to which the coupon has been dissolved, determines the corrosivity of the waste.

3.0 INTERFERENCES

In laboratory tests, such as this one, corrosion of duplicate coupons is usually reproducible to within 10%. However, large differences in corrosion rates may occasionally occur under conditions where the metal surfaces become passivated. Therefore, at least duplicate determinations of corrosion rate should be made.

4.0 APPARATUS AND MATERIALS

An apparatus consisting of a two necked 1-L flask with 24/40 and 45/50 ground glass joints, a reflux condenser with 45/50 joint, a thermometer with a 24/40 joint, a temperature regulating device, a mantle heating device and a glass specimen support system. The method of supporting the coupons has been designed to insulate the coupons from each other physically and electrically and to insulate the coupons from any metallic container or other device used in the test.

A circular SAE 1020 steel coupon of about 3.75 cm (1.5 in.) diameter (D) with a thickness (t) of approximately 0.32 cm (0.125 in.) and a 0.80-cm (0.4-in.) diameter (d) hole for mounting is used. The total surface area (A) of the circular coupon specimen is given by the following equation:

$$A = 3.14/2(D^2-d^2) + (t)(3.14)(D) + (t)(3.14)(d)$$

If the hole is completely covered by the mounting support, the last term in the equation, (t)(3.14)(d) can be omitted. Measure coupons carefully to permit calculation of the exposed areas to an accuracy of $\pm$1%. The minimum ratio of volume of waste to area of the metal coupon to be used in this test is 40 mL/cm^2.

Prepare coupons by chemical treatment, scrubbing with bleach-free scouring powder, followed by rinsing in distilled water and then in acetone or methanol, and finally by air-drying. After final cleaning, the coupons are weighed and stored in a desiccator until used.

5.0 REAGENTS

20% Sodium hydroxide (NaOH). Dissolve 200-g NaOH in 800 mL Type II water.
Concentrated Hydrochloric acid (HCl).
Methanol, Acetone and Methylene chloride.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

All samples should be collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

7.0 PROCEDURE

Assemble the test apparatus and fill the container with the appropriate amount of waste. Begin agitation at a rate sufficient to ensure that the liquid is kept well mixed and homogeneous. Using the heating device, bring the temperature of the waste to 55°C (130°F). An accurate rate of corrosion is not required; only a determination as to whether the rate of corrosion is less than or greater than 6.35 mm per year is required. A 24-hr test period should be ample to determine whether or not the rate of corrosion is >6.35 mm per year.

In order to determine accurately the amount of material lost to corrosion, the coupons have to be cleaned and weighed before and after immersion in the waste. The cleaning procedure employed is chemical treatment using acetone, methanol and methylene chloride in succession. The effect of the chemical cleaning procedure in removing sound metal is determined by using a blank (i.e., a coupon that has not been exposed to the waste). The blank is cleaned along with the test coupon and its weight loss subtracted from that calculated for the test coupons.

8.0 CALCULATIONS

The weight loss (WL) in milligrams of the coupon, its surface area (A) in square centimeters and the length of time (T) in hours employed for the corrosion test are used to calculate corrosion rate in millimeters per year (mmpy). Use of weight loss as a measure of corrosion requires making the assumption that all weight loss has been due to generalized corrosion and not localized pitting. The following formula is used:

$$\text{Corrosion rate (mmpy)} = \frac{WL * 11.145}{A * T}$$

8.0 QUALITY CONTROL

Analyze one sample per batch of 20 or less in duplicate.

9.0 REFERENCES

9.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 6, Properties, December 1996.
9.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 7, section 7.2.2(1)(a), Characteristics Introduction and Regulatory Definitions, December 1996.
9.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 1010, Corrosivity toward Steel.
9.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 1010A, Corrosivity toward Steel, Update IIIB.
9.5 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.
9.6 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.
9.7 AML Standard Operating Procedures, Q-Water-RC.
9.8 AML Standard Operating Procedures, Q-WasteMgmt-RD.

STANDARD OPERATING PROCEDURES

No. W-Flashpoint-CC-RC

IGNITABILITY - FLASHPOINT DETERMINATION BY CLOSED-CUP TESTER (SW-846 Method 1020B)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

IGNITABILITY - FLASHPOINT DETERMINATION BY CLOSED-CUP TESTER (SW-846 Method 1020B)

1.0 SCOPE AND APPLICATION

This standard operating procedure is based on SW-846 Method 1020A, 1010, ASTM D-3278-78, Test Method for Flash Point of Liquids by Setaflash Closed Cup Tester and ASTM D 93-80, Test Methods for Flash Point by Pensky-Martens Closed Tester. These methods use closed-cup testers to determine the flash point of solids and liquids including those that tend to form a surface film under test conditions. Liquids containing non-filterable, suspended solids will also be tested using this SOP. The procedure may be used to determine whether a material will or will not flash at a specified temperature (e.g. room temperature) or to determine the finite temperature at which a material will flash up to 100°C.

2.0 SUMMARY OF METHOD

The sample is heated at a slow, constant rate and a small flame is directed into the cup at regular intervals. The flash point is the lowest temperature at which application of the test flame ignites the vapor above the sample.

3.0 SAMPLE HANDLING AND PRESERVATION

Samples will be stored at 4°C in a closed jar. Samples with flashpoint lower than room temperature will be loaded directly into sample cups without allowing them to equilibrate to room temperature.

4.0 INTERFERENCES

Liquids that tend to form a surface film may yield erroneous results with Setaflash testers. These samples will be tested with Pensky-Martens Closed Cup Testers.

5.0 APPARATUS

Koehler Model K-14600 closed cup tester.

6.0 REAGENTS

Diisobutylketone (DIBK) and p-Xylene. Prepare a standard containing DIBK and p-Xylene (75:25 v/v).

7.0 PROCEDURES

Verify that the thermometers that read sample and bath temperatures are working correctly. Verify that the heating bath contains sufficient water to keep the sample cup completed surrounded by the bath. Open the sample cup and introduce 2 ml of the

standard. Connect the gas line to the tester and light the flame. Raise the temperature of the bath at the rate of 10°F /min. until the bath temperature is approximately 5°F below the specification temperature (102°F for the DIBK/Xylene mixture or 79°F for p-Xylene). Lower the rate of heating by gently adjusting the rate of heating to approx. 4°F/min. Apply the test flame by turning the knob towards you and note whether the sample flashes or not at 15 second intervals. Record the minimum temperature at which the sample flashes. The tester is functioning correctly, if the observed flashpoint is within the limits shown below:

Standard	Expected flashpoint (°F)	Limits
DIBK/p-Xylene (75:25 v/v)	104	99 to 109°F
p-Xylene	79	76 to 83°F

Proceed with unknown sample analysis if the observed flashpoint is within the limits shown above. Otherwise, check the accuracy of the thermometers, integrity of the standards, etc. Take appropriate corrective action to restore tester to proper operating conditions.

Introduce sufficient sample (approx. 5 g or 5 ml) directly into a clean sample cup, which has been brought to either room temperature or below the expected flash point, whichever is lower. The temperature of the apparatus is raised at the rate of 10°F/min. by appropriate adjustment of the temperature dial. After each minute, a test flame is applied inside the cup and note is taken as to whether the test sample flashes or not. If the sample flashes, record the temperature range for the flash and cool the cup to approximately 10° below the flash temperature. Discard the sample and load the cup with fresh sample. Raise the temperature at the rate of 4°F/min testing for flash every 15 seconds. Record the actual flash temperature to an accuracy of 1°F. If a repeat test is necessary, a fresh sample should be used. Stop flashpoint measurements at 210°F.

8.0 CALCULATIONS

Calculations are not required for this physical measurement. Report the flashpoint in°F. If the sample flashes at room temperature, report the flashpoint as <RT°F, where RT is the actual numerical value of the observed flash temperature. If the sample does not flash at 210°F, report the flashpoint as >210°F.

9.0 QUALITY CONTROL

Sample cups must be kept clean, making sure that they do not contain flammable solvents. If solvents are used to clean the sample cup, it must be dried thoroughly prior to use. A minimum of one control standard should be analyzed with each batch of 20 samples. Samples that flash in the 135 to 145°F ranges should be reanalyzed to determine the precision and accuracy of the measurement.

10.0 REFERENCES

10.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 6, Properties, December 1996.

10.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 7, section 7.2.2(1) (a), Characteristics Introduction and Regulatory Definitions, December 1996.

10.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 1020A, Closed-cup Method for Determining Ignitability.

10.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 1020B, Small Scale Closed-cup Method for Determining Ignitability, Update IIIB.

10.5 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.

10.6 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

10.7 AML Standard Operating Procedures, Q-Water-RC.

10.8 AML Standard Operating Procedures, Q-WasteMgmt-RD.

STANDARD OPERATING PROCEDURES

W-Ignitability-RC

IGNITABILITY
(SW-846 Chapter 7)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

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Laboratory Director, Analytical Management Laboratories Date

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Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

IGNITABILITY (SW-846 Chapter 7)

1.0 Scope and Application

1.1 This SOP is used to describe methods and procedures that should be followed to determine the hazardous characteristic of ignitability in waste samples. The regulatory background of this characteristic is summarized, and the regulatory definition of ignitability is presented.

1.2 The two testing methods associated with this characteristic, SW-846 Methods 1010 and 1020 are included in Chapter 8 of the SW-846 document.

1.3 The objective of the ingnitability characteristic is to identify wastes that either present fire hazards under routine storage, disposal and transportation or are capable of severely exacerbating a fire once started.

2.0 Summary of the Guidance Document

2.1 Regulatory Definition - The following definitions have been taken nearly verbatim from the RCRA regulations (40 CFR 261.21) and the DOT regulations (49 CFR 173.300 and 173.151)

2.2 Characteristic of Ignitability - A solid waste exhibits the characteristic of ignitability if a representative sample of the waste has any of the following properties:

2.2.1 It is a liquid, other than an aqueous solution, containing < 24% alcohol by volume, and it has a flash point < 60°C (140°F).

2.2.2 Flashpoint is determined by a Pensky-Martens Closed Cup Tester, using the test method specified in ASTM Standard D-93-79 or D-93-80, or a Setaflash Closed Cup Tester, using the test method specified in ASTM standard D-3278-78, or as determined by an equivalent test method approved by the Administrator under the procedures set forth in sections 260.20 and 260.21 (ASTM standards are available from ASTM, 1916 Race Street, Philadelphia, PA 19103.)

2.2.3 The waste is not a liquid and is capable, under standard temperature and pressure, of causing fire through friction, absorption of moisture, or spontaneous chemical changes and, when ignited, burns so vigorously and persistently that it creates a hazard.

3.0 Procedure

Refer to AML SOP on flashpoint determination for additional details.

4.0 REFERENCES

4.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 6, Properties, December 1996.

4.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 7, section 7.2.2(1) (a), Characteristics Introduction and Regulatory Definitions, December 1996.
4.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 1010, Pensky-Martens Closed-cup Method for Determining Ignitability, Update IIIB.
4.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 1020A, Closed-cup Method for Determining Ignitability.
4.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 1020B, Small Scale Closed-cup Method for Determining Ignitability, Update IIIB.
4.6 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.
4.7 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.
4.8 AML Standard Operating Procedures, Q-Water-RC.
4.9 AML Standard Operating Procedures, Q-WasteMgmt-RD.

STANDARD OPERATING PROCEDURES

No. W-PaintFilter-RC

Paint Filter Liquids Test
(EPA SW-846 9095B)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

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Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Paint Filter Liquids Test (EPA SW-846 9095B)

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) is based on SW-846 Method 9095B, which is used to determine the presence of free liquids in a representative sample of waste for determining compliance with 40 CFR 264.314 and 265.314.

2.0 SUMMARY OF METHOD

2.1 A predetermined amount of material is placed in a paint filter. If any portion of the material passes through and drops from the filter within the 5-min test period, the material is deemed to contain free liquids.

3.0 SAMPLE HANDLING AND PRESERVATION

3.1 **S**amples should be collected in accordance with an approved sampling and analysis plan, prepared in accordance with the directions in Chapter Nine SW-846, Revision 1, December 1996.
3.2 Samples should be stored at 4°C in a closed jar.
3.3 A 100-mL or 100-g representative sample is required for the test.

4.0 INTERFERENCES

4.1 Filter media were observed to separate from the filter cone on exposure to alkaline materials. This development causes no problem if the sample is not disturbed.
4.2 Temperature can affect the test results if the test is performed below the freezing point of any liquid in the sample. Tests must be performed above the freezing point and can, but are not required to, exceed room temperature of 25° C.

5.0 APPARATUS

5.1 Conical paint filter: Mesh number 60 ± 5% (fine meshed size), available at local hardware stores and paint stores such as Sherwin-Williams and Glidden.
5.2 Glass funnel
5.3 Ring stand and ring, or tripod.
5.4 100-mL graduated cylinder or beaker.

6.0 REAGENTS

6.1 None.

7.0 PROCEDURES

7.1 Assemble test apparatus consisting of a ring stand, a funnel equipped with a paint filter (see Note #1) and a beaker or volumetric cylinder under the stem of the funnel.

7.2 Place a 100 g or 100 ml representative sample in the filter. If the sample is measured volumetrically, then it should lack major air spaces or voids. Allow sample to drain for 5 min into the graduated cylinder. If any portion of the test material collects in the graduated cylinder in the 5-min period, then the material is deemed to contain free liquids for purposes of 40 CFR 264.314 and 265.314.

7.3 Record the amount of free liquid five minutes after sample was leaded into the apparatus. If no free liquids are collected, record the volume collected as 0.0 ml and characterize the waste as passing this test. If any portion of the waste contains free liquids (see Note #2), the entire sample is considered to have free liquids.

7.4 ***Note #1: A*** funnel may be used to provide support for the paint filter. If the sample is of such light bulk density that it overflows the filter, then the sides of the filter can be extended upward by taping filter paper to the inside of the filter and above the mesh. Settling the sample into the paint filter may be facilitated by lightly tapping the side of the filter as it is being filled. If the paint filter, with the waste, cannot sustain its weight on the ring stand, then a fluted glass funnel or glass funnel with a mouth large enough to allow at least 1 inch of the filter mesh to protrude should be used to support the filter. The funnel should be fluted or have a large open mouth in order to support the paint filter yet not interfere with the movement, to the graduated cylinder, of the liquid that passes through the filter mesh.

7.5 ***Note #2: If*** it is not possible to obtain a sample of 100 ml or 100 g that is sufficiently representative of the waste, use larger size samples in multiples of 100 ml or 100 g, i.e., 200, 300, 400 ml or g. However, when larger samples are used, divide the sample into 100-mL or 100-g portions and test each portion separately. In order to assure uniformity and standardization of the test, material such as sorbent pads or pillows which do not conform to the shape of the paint filter, should be cut into small pieces and poured into the filter. Sample size reduction may be accomplished by cutting the sorbent material with scissors, shears, knife, or other such device so as to preserve as much of the original integrity of the sorbent fabric as possible. Sorbents enclosed in a fabric should be mixed with the resultant fabric pieces. The particles to be tested should be reduced smaller than 1 cm (i.e., should be capable of passing through a 9.5 mm (0.375 inch) standard sieve). Grinding sorbent materials should be avoided as this may destroy the integrity of the sorbent and produce many "fine particles" which would normally not be present. For brittle materials larger than 1 cm that do not conform to the filter, light crushing to reduce oversize particles is acceptable if it is not practical to cut the material. Materials such as clay, silica gel, and some polymers may fall into this category.

8.0 CALCULATIONS

8.1 None.

8.2 Report the results as a pass/fail test. Samples with free liquids fail the test.

9.0 QUALITY CONTROL

9.1 Duplicate samples should be analyzed on a routine basis. Select samples that fail to perform lab duplicates.

9.2 Record volume of free liquids collected for each analysis.

10.0REFERENCES

10.1Test Methods for Evaluating Solid Waste, Volume 1C: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9095A, Paint Filter Liquids Test, December 1986.

10.2Test Methods for Evaluating Solid Waste, Volume 1C: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9095B, Paint Filter Liquids Test, Update IIIb.

10.3Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 6, Properties, December 1996.

10.4Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 7.

10.5EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.

10.6Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

10.7AML Standard Operating Procedures, Q-Water-RC.

10.8AML Standard Operating Procedures, Q-WasteMgmt-RD.

10.9AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

STANDARD OPERATING PROCEDURE

No. W-Reactivity-RC

REACTIVITY
REACTIVE CYANIDE AND SULFIDE
(SW-846 Chapter 7)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

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Laboratory Director, Analytical Management Laboratories Date

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Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

REACTIVITY
REACTIVE CYANIDE AND SULFIDE
(SW-846 Chapter 7)

1.0 SCOPE AND APPLICATION

This AML standard operating procedure (SOP) to determine reactivity is based on SW-846, Chapter 7.3, "Reactivity", which provides regulatory definitions and an introduction to hazardous characteristics. A solid waste is defined as exhibiting the characteristics of reactivity if a representative sample of the waste has any of the following properties:

1.1 It is normally unstable and readily undergoes violent change without detonating.
1.2 It reacts violently with water.
1.3 It forms potentially explosive mixtures with water.
1.4 When mixed with water, it generates toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.
1.5 It is a cyanide or sulfide bearing waste which, when exposed to pH conditions between 2 and 12.5, can generate toxic gases, vapors, or fumes in quantities sufficient to present a danger to human health or the environment. The presence of reactive cyanide and sulfide can be determined using interim guidance contained in chapters 7.3.3 and 7.3.4 of SW-846. A waste will be considered to be reactive if it contains total releasable cyanide greater than 250 mg HCN/kg waste or total releasable sulfide greater than 500-mg H2S/kg waste.
1.6 It is capable of detonation or explosive reaction if it is subjected to a strong initiating source or if heated under confinement.
1.7 It is readily capable of detonation or explosive decomposition or reaction at standard temperature and pressure.
1.8 It is a forbidden explosive, as defined in 49 CFR 173.51, or a Class A explosive, as defined in 49 CFR 173.53, or a Class B explosive, as defined in 49 CFR 173.88.

The scope of this SOP includes performing inspections and visual observations to determine reactivity as outlined in items 1.1 to 1.4 and 1.7 above and to provide detailed procedures to perform item 5. AML is not equipped to perform tests to determine properties shown in items 1.6 and 1.8 above. These properties (Items 1.6 and 1.8) will not be determined by AML.

2.0 SUMMARY OF METHOD

2.1 A visual inspection of the sample and a qualitative determination of its reactivity with water are initially performed. Rate of HCN and/or H2S release under mildly acidic conditions is determined only if waste contains either detectable or significant levels of total cyanide and/or sulfide, which are determined using appropriate test methods (e.g. SW-846 Method 9010B for cyanide and SW-846 Method 9030B for sulfide). Procedures for determining reactive cyanide and sulfide are included in this SOP.
2.2 An aliquot of mild acid is added to a fixed weight of waste in a closed system for a fixed amount of time. The generated gas (HCN or H2S) is swept into a scrubber. The

contents of the scrubber are tested for cyanide and sulfide using appropriate SOPs.

3.0 SAMPLE HANDLING AND PRESERVATION

3.1 Samples containing, or suspected of containing, sulfide or a combination of sulfide and cyanide wastes should be collected with a minimum of aeration. To the extent possible, the sealed sample bottle should be filled completely devoid of headspace. Analysis should commence as soon as possible, and samples should be kept in a cool, dark, place until analysis begins.

3.2 It is suggested that samples of cyanide wastes be tested as quickly as possible. Although they can be preserved by adjusting the sample pH to 12 with strong base, this will cause dilution of the sample, increase the ionic strength, and, possibly, change other physical or chemical characteristics of the waste which may affect the rate of release of the hydrocyanic acid. Storage of samples should be under refrigeration and in the dark. Testing should be performed in a ventilated hood.

4.0 INTERFERENCES

Interferences are undetermined.

5.0 APPARATUS

For Cyanide and Sulfide determinations:

Round-bottom flask - 500-mL, three-neck, with 24/40 ground-glass joints.
Gas scrubber - 50 mL calibrated scrubber
Stirring apparatus - To achieve approximately 30 rpm. This may be either a rotating magnet and stirring bar combination or an overhead motor-driven propeller stirrer.
Additions funnel - With pressure-equalizing tube and 24/40-ground glass joint and Teflon sleeve.
Flexible tubing - For connection from nitrogen supply to apparatus.
Water-pumped or oil-pumped nitrogen gas - With two-stage regulator.
Rotometer - For monitoring nitrogen gas flow rate.
Analytical balance - capable of weighing to 0.001 g.

6.0 REAGENTS

Reagents required for the distillation part of the procedure are described in this SOP. The colorimetric determination of cyanide and sulfide are not included in this SOP.

Reagent water.
Reagent grade inorganic chemicals. See below:
(a) Sulfuric acid (0.01N), H_2SO_4. Add 2.8-mL concentrated H_2SO_4 to organic-free reagent water and dilute to 1 L. Withdraw 100-mL of this solution and dilute to 1-L to make the 0.01N H_2SO_4.

(b) Cyanide reference solution, (1000 mg/L). Dissolve approximately 2.5 g of KOH and 2.51 g of KCN in 1 liter of organic-free reagent water. Standardize with 0.0192N AgNO3. Cyanide concentration in this solution should be 1 mg/mL.
© Sodium hydroxide solution (1.25N). Dissolve 50 g of NaOH in reagent water and dilute to 1 liter with organic-free reagent water.
(d) Sodium hydroxide solution (0.25N). Dilute 200 mL of 1.25N sodium hydroxide solution (Step 5.5) to 1 liter with organic-free reagent water.
(e) Silver nitrate solution (0.0192N). Prepare by crushing approximately 5 g of AgNO3 crystals and drying to constant weight at 40°C. Weigh 3.265 g of dried AgNO3, dissolve in organic-free reagent water, and dilute to 1 liter.
(f) Sulfide reference solution: Dissolve 4.02 g of $Na_2S \cdot 9H_2O$ in 1.0 L of organic-free reagent water. Dilute this solution containing 570-mg/L hydrogen sulfide to cover the analytical range required (100-570 mg/L).

7.0 PROCEDURES

Add 50-mL of 0.25N NaOH solution to a calibrated scrubber and dilute with organic-free reagent water to obtain an adequate depth of liquid. Close the system and adjust the flow rate of nitrogen, using the rotometer. Flow should be 60 mL/min. Add 10 g of the waste to be tested to the system. With the nitrogen flowing, add enough 0.01N sulfuric acid to fill the flask half full. Start the 30-minute test period. Begin stirring while the acid is entering the round-bottom flask. The stirring speed must remain constant throughout the test. The stirring should not be fast enough to create a vortex. After 30 minutes, close off the nitrogen and disconnect the scrubber.

Adjust the total volume of the distillate in the scrubber to 100-mL. Use this distillate for both cyanide and sulfide analysis using appropriate SOPs. Determine cyanide using the colorimetric procedure described in the SOP "Cyanide-SOP Midi-Still Distillation". Determine sulfide using the colorimetric procedure described in the SOP "Sulfide-Method 376.2 Colorimetric- methylene blue".

8.0 CALCULATIONS

Determine the specific rate of release of HCN or H2S, using the following parameters:

X = Concentration of HCN or H2S in diluted scrubber solution (mg/L)
(This is obtained from cyanide or sulfide analysis)
L = Volume of solution in scrubber (L)
W = Weight of waste used (kg)
S = Time of measurement (sec.) = Time N_2 stopped - Time N_2 started

$$R = \text{specific rate of release (mg/kg/sec.)} = \frac{X \cdot L}{W \cdot S}$$

Total releasable HCN or H2S (mg/kg) = R x S

9.0 QUALITY CONTROL

Reactive or total releasable cyanide (or sulfide) shall not be greater than total cyanide (or sulfide). Repeat reactive cyanide analysis if disparate results are obtained. Perform duplicate and spike analysis for cyanide and sulfide on the distillates.

10.0REFERENCES

10.1Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 6, Properties, December 1996.
10.2Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 7, section 7.2.2(1)(a), Characteristics Introduction and Regulatory Definitions, December 1996.
10.3Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9010C, Total and Amenable Cyanide: Distillation, Update IIIb.
10.4Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9012B, Total and Amenable Cyanide (Automated Colorimetric, with Off-line Distillation), Update IIIB.
10.5Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Sulfide, Method 376.1 (Titrimetric, Iodine), Editorial Revision, 1978.
10.6Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Sulfide (Iodometric), Method 4500-S2-A and 4500-S2-F.
10.7Test Methods for Evaluating Solid Waste, Volume 1C: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9034, Titrimetric Procedure for Acid-soluble and Insoluble sulfides, December 1996.
10.8EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.
10.9Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.
10.10 AML Standard Operating Procedures, Q-Water-RC.
10.11 AML Standard Operating Procedures, Q-WasteMgmt-RD.
10.12 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
10.13 AML Standard Operating Procedures, Q-Controlcharts-RC.

STANDARD OPERATING PROCEDURES

No. W-SPLP-RC

SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SPLP, SW-846 1312)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

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Laboratory Director, Analytical Management Laboratories Date

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Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
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SYNTHETIC PRECIPITATION LEACHING PROCEDURE
(SPLP, SW-846 1312)

1.0 Scope and Application

This SOP is used to prepare leachates in accordance with sw-846 Method 1312, the synthetic precipitation leaching procedure (SPLP). This SOP is similar to the SOP, which is based on SW-846 method 1311, toxicity characteristics leaching procedure (TCLP). These SOPs differ in the nature of extraction fluids used for the leaching procedure. The leachates prepared in accordance with this procedure are subsequently analyzed in accordance with other applicable methods to determine the concentration of parameters such as volatile organics (VOCs), semivolatile organics (SVOCs), organochlorine pesticides (Pest), chlorophenoxy herbicides (Herb) and metals. The leachates from waste and wastewater are analyzed to determine the mobility of organic and inorganic contaminants under certain conditions.

2.0 Summary of the Method

Waste is either simply filtered for liquid wastes (<0.5% solids) or extracted with a leaching fluid that is a function of the alkalinity of the waste. The amount of extraction fluid used is twenty times the weight of the solid phase (2000-ml fluid for 100 g of waste). Leachates are separated from the waste by filtration through a 0.6 to 0.8 µm glass fiber filter. For VOCs analysis, extraction and filtration are performed using zero head space extractors (ZHE).

3.0 Sample Handling and Preservation

Minimum sample size required for complete TCLP analysis of solid samples is 400 g. Store unpreserved samples at 4°C unless it results in irreversible changes to the waste. After leaching the extracts (leachates) are preserved in accordance with the test to be performed and stored at 4°C. The following holding times are applicable for leaching and analysis of extracts:

Test Parameter	Leaching Procedure (from sample collection)	Extraction/Digestion of the leachates (from 1312 leachate prep.)	Instrumental Analysis (from prep. of extracts)
Mercury	28 days	NA	28 days total
All other metals	180 days	NA	180 days total
VOCs	14 days	NA	14 days
SVOCs	14 days	7 days	40 days
Pesticides	14 days	7 days	40 days
Herbicides	14 days	7 days	40 days

Holding times for leaching procedures are calculated from the date of waste or wastewater sample collection date. The holding times for leachate sample preparation (extraction or digestion as applicable) are calculated from the date of collection of the filtered leachate. The holding times for instrumental analysis are calculated from the date of the sample preparation with the exception of metals, which is from the date of leaching procedure.

4.0 Interferences

Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

5.0 Apparatus

Agitation apparatus, an end-over-end rotating (30±2 rpm) tumbler
2.0 L glass bottles with screw caps for organics (and metals, if necessary) extraction
2.0 L plastic bottles with screw caps for metals only
Whatman 0.7 µm glass fiber filter
pH meter
200-ml beakers, stir bar, watch glass

6.0 Reagents

Use only analytical reagent grade products including ASTM Type II water unless otherwise specified.
ASTM Type II water
ASTM Type II water that passed through carbon for VOCs analysis
Concentrated sulfuric acid
Concentrated nitric acid
1.0 N sodium hydroxide (NaOH). Dissolve 40.0 g NaOH in one liter of reagent water.
1.0 N nitric acid. (HNO_3) Dilute con. Nitric acid to the required strength.
1.0 N hydrochloric acid (HCl). Dilute con. Hydrochloric acid to the required strength.

Sulfuric acid/nitric acid (60/40 weight percent mixture) H_2SO_4/HNO_3. Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid. If preferred, a more dilute H2SO4/HNO_3 acid mixture may be prepared and used in the following steps, making it easier to adjust the pH of the extraction fluids.

Extraction fluids:

Extraction fluid #1: This fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids (or a suitable dilution) to reagent water until the pH is 4.20 ± 0.05. The fluid is used to determine the leachability of soil from a site that is east of the Mississippi River, and the leachability of wastes and wastewaters. NOTE: Solutions are unbuffered and exact pH may not be attained.

Extraction fluid #2: This fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids (or a suitable dilution) to reagent water until the pH is 5.00 ± 0.05. The fluid is used to determine the leachability of soil from a site that is west of the Mississippi River.

Extraction fluid #3: This fluid is reagent water and is used to determine cyanide and volatiles leachability.

NOTE: These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

7.0 Procedures

(A) Sample Characterization and Selection of Extraction Fluid:

Perform preliminary 1312 evaluations on a minimum 100-g aliquot of sample. This aliquot may not actually undergo 1312 extraction. These preliminary evaluations include: (1) determination of the percent solids; (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration; and (3) determination of whether the solid portion of the waste requires particle size reduction.

Document observations in the STLP worksheet No. 1. Record date, project number and analyst initials at the top of the worksheet and the sample numbers at appropriate columns. Check appropriate box for sample description. Pressure filtration is not required if the sample will obviously yield no free liquid when subjected to pressure filtration. Notify Project Manager if the sample appears to be muti-phasic and complete section B by pressure-filtering 100 g sample aliquots to a maximum of 50 psi pressure and recording the weight of solids and filtrates. Perform filtration in accordance with SW-846 method 1312 criteria. Material in the filter holder (solid, sludge or unfilterable liquid) is defined as the solid phase. Determine percent solids as weight of solid phase divided by the total initial sample weight expressed as percentage.

Perform size reduction of the solids if necessary by crushing, cutting or grinding so that the resulting material can pass through a 9.5-mm (0.375-inch) sieve.

Determine the appropriate extraction fluid that is necessary by performing steps described in section C of the STLP Worksheet No. 1 and by completing each item in this section. Notify project manager that additional information about the geographical origin (east or west of Mississippi River, etc.) is required for extraction fluid selection.

Determination of appropriate extraction fluid: Use the following table to determine the appropriate extraction fluid. For soils, if the sample is from a site that is east of the Mississippi River, extraction fluid #1 should be used. If the sample is from a site that is

west of the Mississippi River, extraction fluid #2 should be used. For wastes and wastewater, extraction fluid #1 should be used. For cyanide-containing wastes and/or soils, extraction fluid #3 (reagent water) must be used because leaching of cyanide-containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

Extraction Fluid #1 Dilute 60/40 H_2SO_4/HNO_3 (pH 4.20±0.05)	**Extraction Fluid #2** Dilute 60/40 H_2SO_4/HNO_3 (pH 5.00±0.05)	**Extraction Fluid #3** Reagent Water
Soil east of Mississippi River	Soil west of Mississippi River	Cyanide containing soil
Wastes		Cyanide containing waste
Wastewater		Volatiles determination in all matrices

(B) Preparation of TCLP Extracts for metals, SVOCs, Pesticides and Herbicides Analysis

Follow instructions shown in STLP Worksheet No. 2 and document data collected for each sample in the appropriate column. Use 100 g of dry solids (step E2) or filter 100 g of mutiphasic waste. (NOTE: A fresh quantity of sample should be filtered if the solid phase was dried to make percent solids measurements documented in STLP Worksheet No. 1). Use twenty times the volume of extraction fluid selected in section D of STLP Worksheet No. 1. (e.g. 2000-ml for 100-g of sample). If filtration is necessary amount of solid waste may be reduced to a quantity less than 100 g. Correspondingly less EF should be used. Store the filtrate in a glass container. Test water miscibility by adding a small aliquot (0.1-ml) to 1 ml of the EF to be used for STLP leachate preparation. If the filtrate is not miscible label it as "STLP Non-Aqueous" along with AML sample numbers. A separate analysis of the filtrate will be necessary.

Quantitatively transfer solid phase along with the filter to the extraction vessel to perform STLP extraction. Close the bottle using a screw-cap lid using Teflon tape, if necessary, for a tight seal. Secure the bottles in the rotary agitation device and tumble them for at 30± 2 rpm for 18± 2 hours. Relieve pressure (potentially from carbon dioxide build-up) from bottles twice after each 30 minutes of initial tumbling. Document start and stop dates and times in section G of the Worksheet. Pressure-filter the required volume of the leachates in to a 2.0-liter container, changing filter papers as necessary to complete the task. The filtrate is the entire STLP Extract for single-phase solid wastes. Measure the pH and the total volume of the filtrate.

Combine the STLP Extract collected above with any aqueous or water-miscible filtrate collected from the waste in step E.3.b of the Worksheet for multi-phasic samples. The combined material is the entire STLP Extract for multi-phasic waste not containing non-aqueous materials. Preserve the STLP Extract by chilling them to 4°C and with

preservatives as applicable for each test to be performed and label them as "STLP Aqueous" labeling each container with appropriate AM Lab sample numbers.

(C) Preparation of STLP Extracts for VOCs Analysis

The initial filtration and STLP extraction are performed in specially designed ZHE vessels. Specialized training will be necessary to set up and operate the ZHE vessels. Contact the Operations Manager prior to performing this procedure for an initial training. Only EF #3 is used to prepare STLP extracts for VOCs analysis. Weight 20.0 g of the waste sample and record the weight in section I of the Worksheet. Charge the ZHE vessel with the sample and filter the liquid phase, collecting it in a Tedlar bag or a small bottle with a septum cap. Charge the ZHE with the required volume (see step I.2) of EF #3 and tumble the vessels as described for the SVOCs STLP extracts preparation. Filter the final extract using pressure and collect the aqueous leachate appropriately without headspace. If the initial and final filtrates are miscible and compatible they can be combined inside the Tedlar bag or other appropriate vessel. Otherwise, the two filtrates should be analyzed separately and the results combined mathematically.

8.0 Calculations

If the initial liquid phase of the waste as obtained in step E.3.b of the Worksheet is either not compatible or immiscible with the filtered liquid from step G.5 of the Worksheet, these liquids are not combined. These liquids collectively are defined as the STLP Extract for this multi-phasic waste. Each liquid is analyzed separately and the results are combined mathematically using SOPs applicable to each test. Inform the Project Manager about the existence of multiple STLP liquid phases for applicable samples.

9.0 Quality Control

For each batch of 20 samples or less, perform the following quality control measurements:
1 method blank-tumble appropriate volumes of the Extraction Fluid only

10.0 References

10.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 6, Properties, December 1996.

10.2Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 7, section 7.2.2(1) (a), Characteristics Introduction and Regulatory Definitions, December 1996.

10.3Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 1311, Toxicity Characteristic Leaching Procedure, Revision 0, July 1992.

10.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 1312, Synthetic Precipitation Leaching Procedure, Revision 0, September 1994.
10.5 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.
10.6 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.
10.7 AML Standard Operating Procedures, Q-Water-RC.
10.8 AML Standard Operating Procedures, Q-WasteMgmt-RD.
10.9 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
10.10 AML Standard Operating Procedures, Q-Controlcharts-RC.
10.11 AML Standard Operating Procedures, O-OCPesticides-RB.
10.12 AML Standard Operating Procedures, O-Herbicides-RB.
10.13 AML Standard Operating Procedures, O-VOCs-GCMS-RB.
10.14 AML Standard Operating Procedures, O-BNA-SVOCs-GCMS-RB.
10.15 AML Standard Operating Procedures, I-ICP-AES-Metals-RB.
10.16 AML Standard Operating Procedures, I-ICP-MS-Metals-RB.

ANALYTICAL MANAGEMENT LABORATORIES
SPLP WORKSHEET No. 1 - Sample Description & Selection of Extraction Fluid

DATE: _____ ANALYST: _____ PROJECT: _____

Laboratory Sample Number				
A. Sample Description				
1. Solid				
2. Liquid				
a. lighter than water				
b. water				
c. heavier than water				
B. Percent Solid Phase				
1. Weight of filter (g)				
2. Weight of subsample				
3. Weight of Filtrate				
4. Weight percent solids (wet)				
5. Weight percent solids (dry)				
6. Volume of initial aqueous filtrate				
7. Volume of initial organic filtrate				
C. Extraction Fluid Determination				
1. Particle size reduction? (yes/no)				
2. Is the sample soil? (yes/no)				
3. Soil east of Mississippi river? (yes/no)				
4. Soil west of Mississippi river? (yes/no)				
5. Is the sample waste? (yes/no)				
6. Is the sample wastewater? (yes/no)				
7. Is sample cyanide containing? (yes/no)				
D. Selection of Extraction Fluid (EF)				
1. EF # 1 if "yes" for (C-1/C-3), C-5, and C-6				
2. EF # 2 if "yes" for (C-1/C-4)				
3. EF # 3 if "yes" for C-7 and for VOCs/ZHE				

ANALYTICAL MANAGEMENT LABORATORIES
SPLP WORKSHEET No. 2 - Determination of Extraction Fluid Volume & Record of Extraction

DATE: _____ ANALYST: _____ PROJECT: _____

Laboratory Sample Number				
E. Determination of Sample Size for SV				
1. Particle Size Reduction? yes/no				
2. Amount of dry solids (100-g min.)				
3. Amount of multi-phasic sample				
a. weight of material				
b. weight of filtrate				
c. weight of solid material				
F. Amount of EF for SVOCs & Metals				
1. for dry solids (20 x sample wt.)				
2. for muti-phasic samples (20x(B2-B3))				
G. Record of SVOC/Metals Extraction				
1. pH of the extraction fluid				
2. Temperature of the room				
3. RPM of the tumbler				
4. Extraction Start Date & Time				
5. Extraction Stop Date & Time				
6. Filtration complete Date & Time				
7. pH of filtrate				
8. Volume of filtrate				
H. Determ. of Sample Size for ZHE				
1. Amount of dry solids				
2. Amount of multi-phasic sample				
I. Amount of EF # 3 for ZHE				
1. for dry solids (20 x sample wt.)				
2. for muti-phasic samples (20 x W) W = Wt. Filtered – Wt. of filtrate				
J. Record of ZHE Extraction				
1. Extraction Start Date & Time				
2. Starting pressure				
3. Extraction Stop Date & Time				
4. x if positive pressure				
5. Filtration complete Date & Time				
6. pH of filtrate				
7. Volume of filtrate				
8. Temperature of the room				
9. RPM of the tumbler				
10. pH of the EF #3 (Type II water)				

STANDARD OPERATING PROCEDURES

W-TCLP-RC

TOXICITY CHARACTERISTICS LEACHING PROCEDURE (TCLP, SW-846 1311A)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

TOXICITY CHARACTERISTICS LEACHING PROCEDURE (TCLP, SW-846 1311A)

1.0 Scope and Application

This SOP is used to prepare leachates in accordance with the toxicity characteristics leaching procedure (TCLP). The leachates prepared in accordance with this procedure are subsequently analyzed in accordance with other applicable methods to determine the concentration of parameters such as volatile organics (VOCs), semivolatile organics (SVOCs), organochlorine pesticides (Pest.), chlorophenoxy herbicides (Herb.) and metals. The leachates from waste and wastewater are analyzed to determine the mobility of organic and inorganic contaminants, the hazardous (TCLP standards) characteristics of a solid waste and/or to determine whether treatment procedures meet standards such as the universal treatment standards (UTS).

2.0 Summary of the Method

Waste is either simply filtered for liquid wastes (<0.5% solids) or extracted with a leaching fluid that is a function of the alkalinity of the waste. The amount of extraction fluid used is twenty times the weight of the solid phase (2000 ml fluid for 100 g of waste). Leachates are separated from the waste by filtration through a 0.6 to 0.8 µm glass fiber filter. For VOCs analysis, extraction and filtration are performed using zero head space extractors (ZHE).

3.0 Sample Handling and Preservation

Minimum sample size required for complete TCLP analysis of solid samples is 400 g. Store unpreserved samples at 4°C unless it results in irreversible changes to the waste. After leaching the extracts (leachates) are preserved in accordance with the test to be performed and stored at 4°C. The following holding times are applicable for leaching and analysis of extracts:

Test Parameter	Leaching Procedure	Extraction/Digestion of the leachates	Instrumental Analysis
Metals except mercury	14 days	28 days	28 days total
All other metals	14 days	6 months	6 months total
VOCs	14 days	NA	14 days
SVOCs	14 days	7 days	40 days
Pesticides	14 days	7 days	40 days
Herbicides	14 days	7 days	40 days

Holding times for leaching procedures are calculated from the date of waste or wastewater sample collection date. The holding times for leachate sample preparation (extraction or digestion as applicable) are calculated from the date of collection of the filtered leachate. The holding times for instrumental analysis are calculated from the date of the sample preparation with the exception of metals, which is from the date of leaching procedure.

4.0 Interferences

Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

5.0 Apparatus

Agitation apparatus, an end-over-end rotating (30±2 rpm) tumbler
2.0 L glass bottles with screw caps for organics (and metals, if necessary) extraction
2.0 L plastic bottles with screw caps for metals only
Whatman 0.7 µm glass fiber filter
pH meter
200-ml beakers, stir bar, watch glass

6.0 Reagents

Use only analytical reagent grade products including Type II water unless otherwise specified.
Glacial acetic acid
1.0 N sodium hydroxide (NaOH). Dissolve 40.0 g NaOH in one liter of reagent water.
1.0 N nitric acid. (HNO_3) Dilute con. Nitric acid to the required strength.
1.0 N hydrochloric acid (HCl). Dilute con. Hydrochloric acid to the required strength.

Extraction Fluid #1 (EF#1): Combine 5.7 ml of glacial acetic acid and 64.3 ml of 1.0 N sodium hydroxide to prepare 1.0 liter of EF#1 with reagent water making up the balance. Scale up as necessary. Measure pH and record it in TCLP worksheet No. 1.If it is not 4.93±0.05 adjust pH with HCl or Na OH as necessary.

Extraction Fluid #2 (EF#2): Dilute 5.7 ml of glacial acetic acid with reagent water to prepare 1.0 liter of EF#2. Scale up as necessary. Measure pH and record it in TCLP worksheet No. 1. If it is not 2.88±0.05 adjust pH with HCl or Na OH as necessary.

7.0 Procedures

(A) Sample Characterization and Selection of Extraction Fluid:

Document observations in TCLP worksheet No. 1. Record date, project number and analyst initials at the top of the worksheet and the sample numbers at appropriate columns. Check appropriate box for sample description. Notify Project Manager if the sample appears to be muti-phasic and complete section B by pressure-filtering 100 g sample aliquots to a maximum of 50 psi pressure and recording the weight of solids and filtrates. Perform filtration in accordance with SW-846 method 1311 criteria. Material in the filter holder (solid, sludge or unfilterable liquid) is defined as the solid phase. Determine percent solids as weight of solid phase divided by the total initial sample weight expressed as percentage.

Perform size reduction of the solids if necessary by crushing, cutting or grinding so that the resulting material can pass through a 9.5-mm (0.375-inch) sieve. Determine the appropriate extraction fluid that is necessary by performing steps described in section C of the TCLP Worksheet No. 1 and by completing each item in this section. Sample (5.0 g) is mixed with water (96.5 ml) so that the resulting slurry contains waste and water in a 1:20 ratio (~100 ml total volume). Use a magnetic stirrer and heat to 50°C if necessary after adding 3.5 ml of 1.0 N HCl for basic wastes. Select and use EF#1 for TCLP extraction if the initial pH after 5 min. (see step C4 in the worksheet) is <5.0. Otherwise, use EF #2.

(B) Preparation of TCLP Extracts for metals, SVOCs, Pesticides and Herbicides Analysis

Follow instructions shown in TCLP Worksheet No. 2 and document data collected for each sample in the appropriate column. Use 100 g of dry solids (step E2) or filter 100 g of mutiphasic waste. (NOTE: A fresh quantity of sample should be filtered if the solid phase was dried to make percent solids measurements documented in TCLP Worksheet No. 1). Use twenty times the volume of extraction fluid selected in section D of TCLP Worksheet No. 1. (e.g. 2000 ml for 100 g of waste). If filtration is necessary amount of solid waste may be reduced to a quantity less than 100 g. Correspondingly less EF should be used. Store the filtrate in a glass container. Test water miscibility by adding a small aliquot (0.1-ml) to 1 ml of the EF to be used for TCLP leachate preparation. If the filtrate is not miscible label it as "TCLP Non-Aqueous" along with AM Lab sample numbers. A separate analysis of the filtrate will be necessary.

Quantitatively transfer solid phase along with the filter to the extraction vessel to perform TCLP extraction. Close the bottle using a screw-cap lid using Teflon tape, if necessary, for a tight seal. Secure the bottles in the rotary agitation device and tumble them for at 30± 2 rpm for 18± 2 hours. Relieve pressure (potentially from carbon dioxide build-up) from bottles twice after each 30 minutes of initial tumbling. Document start and stop dates and times in section G of the Worksheet. Pressure-filter the required volume of the leachates in to a 2.0-liter container, changing filter papers as necessary to complete the task. The filtrate is the entire TCLP Extract for single-phase solid wastes. Measure the pH and the total volume of the filtrate.

Combine the TCLP Extract collected above with any aqueous or water-miscible filtrate collected from the waste in step E.3.b of the Worksheet for multi-phasic samples. The combined material is the entire TCLP Extract for multi-phasic waste not containing non-aqueous materials. Preserve the TCLP Extract by chilling them to 4°C and with preservatives as applicable for each test to be performed and label them as "TCLP Aqueous" labeling each container with appropriate AM Lab sample numbers.

(C) Preparation of TCLP Extracts for VOCs Analysis

The initial filtration and TCLP extraction are performed in specially designed ZHE vessels. Specialized training will be necessary to set up and operate the ZHE vessels. Contact the Operations Manager prior to performing this procedure for an initial training. Only EF #1 is used to prepare TCLP extracts for VOCs analysis. Weight 20.0 g of the waste sample and record the weight in section I of the Worksheet. Charge the ZHE vessel with the sample and filter the liquid phase, collecting it in a Tedlar bag or a small bottle with a septum cap. Charge the ZHE with the required volume (see step I.2) of EF #1 and tumble the vessels as described for

the SVOCs TCLP extracts preparation. Filter the final extract using pressure and collect the aqueous leachate appropriately without headspace. If the initial and final filtrates are miscible and compatible they can be combined inside the Tedlar bag or other appropriate vessel. Otherwise, the two filtrates should be analyzed separately and the results combined mathematically.

8.0 Calculations

If the initial liquid phase of the waste as obtained in step E.3.b of the Worksheet is either not compatible or immiscible with the filtered liquid from step G.5 of the Worksheet, these liquids are not combined. These liquids collectively are defined as the TCLP Extract for this multi-phasic waste. Each liquid is analyzed separately and the results are combined mathematically using SOPs applicable to each test. Inform the Project Manager about the existence of multiple TCLP liquid phases for applicable samples.

9.0 Quality Control

For each batch of 20 samples or less, perform the following quality control measurements:
1 method blank-tumble appropriate volumes of the Extraction Fluid only
1 sample of each matrix and/or waste type in triplicate for matrix spike (MS) and MS duplicate.

10.0 References

10.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 6, Properties, December 1996.
10.2Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 7, section 7.2.2(1) (a), Characteristics Introduction and Regulatory Definitions, December 1996.
10.3Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 1311, Toxicity Characteristic Leaching Procedure, Revision 0, July 1992.
10.4Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 1312, Synthetic Precipitation Leaching Procedure, Revision 0, September 1994.
10.5EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.
10.6Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.
10.7AML Standard Operating Procedures, Q-Water-RC.
10.8AML Standard Operating Procedures, Q-WasteMgmt-RD.
10.9AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
10.10 AML Standard Operating Procedures, Q-Controlcharts-RC.
10.11 AML Standard Operating Procedures, O-OCPesticides-RB.
10.12 AML Standard Operating Procedures, O-Herbicides-RB.

10.13 AML Standard Operating Procedures, O-VOCs-GCMS-RB.
10.14 AML Standard Operating Procedures, O-BNA-SVOCs-GCMS-RB.
10.15 AML Standard Operating Procedures, I-ICP-AES-Metals-RB.
10.16 AML Standard Operating Procedures, I-ICP-MS-Metals-RB.

ANALYTICAL MANAGEMENT LABORATORIES
TCLP WORKSHEET No. 1 - Sample Description & Selection of Extraction Fluid

DATE: _____ ANALYST: _____ PROJECT: _____

Laboratory Sample Number				
A. Sample Description				
1. Solid				
2. Liquid				
a. lighter than water				
b. water				
c. heavier than water				
B. Percent Solid Phase				
1. Weight of filter (g)				
2. Weight of subsample				
3. Weight of Filtrate				
4. Weight percent solids (wet)				
5. Weight percent solids (dry)				
6. Volume of initial aqueous filtrate				
7. Volume of initial organic filtrate				
C. Extraction Fluid Determination				
1. Particle size reduction? yes/no				
2. Sample weight, X if 5.0 ± 0.1 g				
3. Volume of water, X if 96.5 ± 1.0 ml				
4. Initial pH, after 5 min. mix time				
5. If pH >5.0, X if 3.5 ml 1N HCl added				
6. X if heated and held at 50C for 10 min				
7. Secondary pH (at room temp.)				
D. Selection of Extraction Fluid (EF)				
1. EF # 1 if pH from C.4 is < 5.0				
2. EF # 2 if pH from C.7 is > 5.0				

ANALYTICAL MANAGEMENT LABORATORIES
TCLP WORKSHEET No. 2 - Determination of Extraction Fluid Volume & Record of Extraction

DATE: _____ ANALYST: _____ PROJECT: _____

Laboratory Sample Number				
E. Determination of Sample Size for SV				
1. Particle Size Reduction? yes/no				
2. Amount of dry solids (100-g min.)				
3. Amount of multi-phasic sample				
a. weight of material				
b. weight of filtrate				
c. weight of solid material				
F. Amount of EF for SVOCs & Metals				
1. for dry solids (20 x sample wt.)				
2. for muti-phasic samples (20x(B2-B3))				
G. Record of SVOC/Metals Extraction				
1. Extraction Start Date & Time				
2. Extraction Stop Date & Time				
3. Filtration complete Date & Time				
4. pH of filtrate				
5. Volume of filtrate				
H. Determ. of Sample Size for ZHE				
1. Amount of dry solids				
2. Amount of multi-phasic sample				
I. Amount of EF # 1 for ZHE				
1. for dry solids (20 x sample wt.)				
2. for muti-phasic samples (20 x W) W = Wt. filtered - Wt. of filtrate				
J. Record of ZHE Extraction				
1. Extraction Start Date & Time				
2. Starting pressure				
3. Extraction Stop Date & Time				
4. x if positive pressure				
5. Filtration complete Date & Time				
6. pH of filtrate				
7. Volume of filtrate				

STANDARD OPERATING PROCEDURES

No. I-Perchlorate-RD

DETERMINATION OF PERCHLORATE BY ION CHROMATOGRAPHY (EPA 314.0 and SW-846 9058)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Quality Assurance Officer, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Analytical Management Laboratories

DETERMINATION OF PERCHLORATE BY ION CHROMATOGRAPHY (EPA 314.0 and SW-846 9056)

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) for perchlorate analysis is based on SW-846 Method 9058, EPA Method 314.0 and ion chromatography methods such as EPA 300.1, and SW-846 Method 9056. This SOP also incorporates instrument manufacturer's recommended procedures.

1.1 This SOP addresses the determination of perchlorate in aqueous samples (reagent water, groundwater, drinking water, wastewater, etc) that may contain high levels of other anions such as chloride, phosphate, and sulfate.

1.2 The method detection limit (MDL), the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero is estimated at 1-ug/L for perchlorate in reagent water samples.

1.2.1 The MDL for a specific matrix may differ from those listed, depending upon the nature of the sample and the specific instrumentation employed.

1.2.2 In order to achieve detection limits stated above, the ion chromatographic system should utilize suppressed conductivity detection, be properly maintained, and must be capable of yielding a baseline with no more than 5 nanoSiemen (nS) noise/drift per minute of monitored response over the background conductivity.

1.3 This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms. Users of the method data should identify data quality objectives prior to analysis. Users of the method must demonstrate the ability to generate acceptable results.

1.4 When this method is used to analyze unfamiliar samples for perchlorate, anion identification should be supported by the use of matrix spike (MS or LFM) and matrix spike duplicate (MSD or LFMD) samples.

1.5 The upper limit of the method is dependent on total anion concentration and may be determined experimentally. These limits may be extended by appropriate dilution. The columns and conditions identified in this SOP bore the greatest tolerance for the highest levels of common inorganic anion interference.

1.6 This SOP is written to comply with the requirements of EPA 314.0. The SW-9058 method requirements are closer to those of SW-846 Method 9056, which describes the ion chromatographic procedure for the analysis of common anions. Strict compliance with this SOP is not required when Method 9058 is specified for sample analysis.

2.0 SUMMARY OF METHOD AND DEFINITIONS

2.1 Approximately 1.0-mL volume of sample is introduced into an ion chromatograph (IC). Perchlorate is separated and measured, using a system comprised of an ion chromatographic pump, sample injection valve, guard column, analytical column,

suppressor device, and conductivity detector. Perchlorate is identified based on its retention time compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a calibration curve generated from known standards.

2.2 EPA Method 314.0 includes a number of strange nuances, numerous acronyms and related definitions. The following definitions have been imported from EPA 314.0 and modified in accordance with SW-846 based definitions and procedures that are commonly used by commercial environmental laboratories:

2.2.1 PERCHLORATE ANALYSIS BATCH - A sequence of samples, which are analyzed within a 30-hour period and include no more than 20 field samples.

2.2.1.1 Every field sample analysis, including both diluted and pretreated field samples, but excluding any MS (LFM) or duplicate field sample analysis which qualify as QC samples, must be applied to the maximum of 20 total field samples permitted in an analysis batch.

2.2.1.2 For projects that require strict compliance with EPA 314.0, an Analysis Batch should also include all required QC samples, which do not contribute to the maximum field sample total of 20. The required QC samples include:

Instrument Performance Check Standard (IPC)
Method Blank (MB) or Laboratory Reagent Blank (LRB)
Initial Calibration Verification (ICV) or Check Standard (ICCS)
Laboratory Control Sample (LCS) or Fortified Blank (LFB)
Matrix Spike (MS) or Laboratory Fortified Matrix (LFB)
Duplicate Sample Analysis (e.g. MSD, field sample duplicate or laboratory sample duplicate)
Continuing Calibration Verification (CCV) or Check Standard (CCCS), when the batch contains more than 10 field samples
Ending CCV or Calibration Check Standard (ECCS)

2.2.1.3 If pretreated samples are analyzed, they should have a separate set of pretreated MB, pretreated LCS and/or LCSD and pretreated MS/MSD. This is in addition to MB, LCS/LCSD, and MS/MSD for untreated samples.

2.2.1.4 Field sample(s), included in the analysis batch, can be reanalyzed following the ending CCV (ECCS) provided the 30 hr time limit for the analysis batch has not expired. The laboratory can reanalyze that sample(s) but must initially conduct a second ICV (ICCS) before the reanalysis and a second ending CCV (ECCS) after the final reanalysis. The second ending CCV (ECCS) must be completed within the 30 hr window.

2.2.2 MATRIX CONDUCTIVITY THRESHOLD (MCT) – The highest permitted conductance of an unknown sample matrix, measured prior to conducting the analysis, which is used to determine when sample matrix dilution or pretreatment is required.

2.2.2.1 The conductance of a sample matrix is proportional to the common anions present in the matrix (which contributes to the level of total dissolved solids [TDS]) which can greatly affect the integrity of this analysis.

2.2.2.2 The value for this threshold (MCT) is dependent on the conditions, hardware, and state of the hardware employed. Consequently, this threshold is not method defined and must be determined by the laboratory during the Initial Demonstration of Capability (IDC) and confirmed in each analysis batch using the Instrument Performance Check (IPC) Solution.

2.2.3 Conductivity - Sample conductance is directly related to the common anion levels in a matrix and its measurement is used to determine if sample pretreatment is required. Conductance measurement is also used to establish and monitor the MCT and represents an objective means of assessing analytical performance when analyzing high conductivity matrices. Conductivity is measured in microSiemens/cm (uS/cm) or microMhos/cm (uMhos/cm) which are considered equivalent terms.

2.2.4 PEAK AREA TO HEIGHT RATIO (A/H) – The ratio of the peak area divided by the peak height which is used as a tool to monitor analytical performance.

2.2.4.1 This ratio is used to establish and monitor the MCT and represents an objective means of assessing analytical performance when analyzing high conductivity matrices.

2.2.4.2 A gradual distortion of the baseline is typically observed in the retention time window for perchlorate as the matrix conductivity increases (consistent with elevated levels of common anions) which will more significantly influence peak height relative to the influence on peak area. As the distortion of the baseline increases, this ratio increases, and the integrity of the measured perchlorate will be compromised.

2.2.5 PEAK GAUSSIAN FACTOR (PGF) - The column efficiency is determined by calculating peak gaussian factor from the chromatographic peak width at half height ($W_{0.50}$) and peak width close to the baseline at one tenth of the height ($W_{0.10}$) using the following equation.

$$PGF = \frac{1.83 * W_{0.50}}{W_{0.10}}$$

2.2.6 TOTAL DISSOLVED SOLIDS (TDS) - Both organic and inorganic constituent which are dissolved in a sample matrix and are not removed by particulate filtration.

3.0 INTERFERENCES

3.1 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in an ion chromatogram. These interferences can lead to false positive results for the target analyte as well as reduced detection limits as a consequence of elevated baseline noise.

3.2 Samples that contain particles larger than 0.45-µm and reagent solutions that contain particles larger than 0.45-µm may damage the instrument columns and flow systems.

3.2.1 All reagent solutions (eluents, external water for ASRS suppressor, etc.) used by the instrument must be filtered through no larger than a 0.45 um nominal pore size membrane or frit to remove particulates and prevent damage to the instrument, columns and flow systems.

3.2.2 Sample filtration must also be employed on every sample prior to analysis. This applies not only to field samples but also to the MB (LRB) and LCS (LFB). The MB and LCS samples function as controls and must be filtered to confirm no bias is attributable to the filtration. Filter the samples through a membrane or frit with no larger than a 0.45 um nominal pore size.

3.2.3 Syringe mounted, cartridge type, filters work well. Filters specifically designed for IC applications should be used.

3.3 Interferences can be divided into three different categories: direct chromatographic coelution, where an analyte response is observed at very nearly the same retention time as the target anion; concentration dependant coelution, which is observed when the response of higher than typical concentrations of the neighboring peak overlap into the retention window of the target anion; and, ionic character displacement, where retention times may significantly shift due to the influence of high ionic strength matrices (high mineral content or hardness) overloading the exchange sites in the column and significantly shortening target analyte's retention times.

3.3.1 A direct chromatographic coelution may be solved by changing columns, eluent strength, modifying the eluent with organic solvents (if compatible with the IC column), changing the detection systems, or selective removal of the interference with pretreatment. Sample dilution will have little to no effect. The analyst should verify that these changes do not induce any negative effect on method performance.

3.3.2 Sample dilution may resolve some of the difficulties if the interference is the result of either concentration dependant coelution or ionic character displacement. However, sample dilution will alter the detection, reporting, and quantitation limits by a proportion equivalent to that of the dilution. Therefore, careful consideration of project objectives should be given prior to performing such a dilution.

3.3.3 Pretreatment cartridges can be effective as a means to eliminate certain matrix interferences. With advances in analytical separator column technology, which employ higher capacity anion exchange resins, the need for these cartridges has been greatly reduced.

3.3.3.1 With any proposed pretreatment, the analyst must verify that the target analyte is not affected by monitoring recovery after pretreatment (e.g. additional MB, LCS/LCSD and MS/MSD on pretreated samples) and that no background contaminants are introduced by the pretreatment.

3.3.3.2 Extreme caution should be exercised in using pretreatment cartridges. Artifacts are known to leach from certain cartridges that can foul the guard and analytical columns causing loss of column capacity indicated by shortened retention times and irreproducible results. Frequently compare your calibration standard chromatograms to those of the column test chromatogram (received when the column was purchased) or use calibration chromatograms generated when the column was initially installed, to insure proper separation and similar response ratios between the target analytes are observed.

3.3.3.3 If MB (LRB) background problems are encountered in the retention time window for perchlorate when these pretreatment cartridges have been employed, increase the initial reagent water rinse of the cartridge to approximately five times the volume specified by the manufacturer.

3.4 Sample matrices with high concentrations of common anions such as chloride, sulfate and carbonate can make the analysis problematic by destabilizing the baseline in the retention time window for perchlorate. This is evidenced by observing a protracted tailing following the initial elution of the more weakly retained anions (chloride, carbonate, and sulfate) which extends into the perchlorate retention time window. These common anion levels can be indirectly assessed by monitoring the conductivity of the matrix. Consequently, all sample matrices must be monitored for conductivity prior to analysis. When the laboratory determined Matrix Conductivity Threshold (MCT) is exceeded, procedures incorporating sample dilution and/or pretreatment should be performed.

3.5 Close attention should be given to the potential for carry over peaks from one analysis that will effect the proper detection of perchlorate in a second, subsequent analysis.

3.5.1 It is the responsibility of the user to confirm that no late eluting peaks have carried over into a subsequent analysis thereby compromising the integrity of the analytical results.

3.5.2 A reagent blank should be analyzed immediately after the analysis of the highest calibration standard to demonstrate that carryover is not a problem. Rinse volumes should be increased (minimum three times the sample loop) between analyses if carryover is suspected.

4.0 APPARATUS AND MATERIALS

4.1 Ion chromatograph, Dionex DX-120, capable of delivering 1 to 5 ml of eluent per minute at a pressure of 1500 to 1700 psi. The chromatograph is equipped with an injection valve, sample loops of varying sizes (25- to 1000-µL), and it is set up with the following components:

4.1.1 Sample Loop - A large sample loop (1.0-mL) can be made using approximately 219 cm (86 inches) of 0.03-inch i.d. PEEK tubing. The exact volume is not critical since all standards and samples will use the same sample loop. However, the volume should be verified to be within 5% of this volume by weighing the sample loop empty, filling the loop with deionized water and re-weighing the loop. Estimate the volume by assuming the density of water at 1.0 mg/µL.

4.1.2 Precolumn, a guard column placed before the separator column to protect the separator column from being fouled by particulates or certain organic constituents (IonPac AG16 4-mm, Dionex P/N 55377). If omitted from the system, the retention times will be shorter.

4.1.3 Separator column, a column packed with low-capacity pellicular anion exchange resin that is styrene divinylbenzene-based has been found to be suitable for resolving perchlorate from other interfering anions (AS16, 4-mm, Dionex P/N 55376). The column should be capable of yielding a symmetrical peak elution with a PGF (See Section 2.2.5) between 0.80 and 1.15.

4.1.4 Suppressor column (ASRS), a column that is capable of converting the eluent and separated anions to their respective acid forms (ASRS Ultra 4-mm, Dionex P/N 53946).

4.1.4.1 Adequate baseline stability should be attained as measured by a combined baseline drift/noise of no more than 5 nS per minute over the background conductivity.

4.1.4.2 The ASRS should be set to perform electrolytic suppression at a current setting of 300-mA using the external water mode. External water should be delivered to the suppressor directly from a pressurized source at a flow rate of 5 mL/min.

4.1.4.3 If the instrument begins to have problems with reduced peak response or asymmetrical perchlorate peaks, the suppressor membranes should be cleaned. As a quick and easy cleaning step, the manufacturer's ASRS "Quickstart" procedure for installing a new ASRS should be followed. If this procedure does not correct the problem, follow the manufacturer's recommended cleaning procedure for removing metal contaminants.

4.1.5 Detector, CD-20, a low-volume, follow-through, temperature-compensated, electrical conductivity cell (approximately 6-µL volume, Dionex CDM-3 standard cell) equipped with a meter capable of reading from 0 to 1,000 µseconds/cm on a linear scale.

4.1.6 Pump, capable of delivering a constant flow of approximately 2 to 5 ml/min throughout the test and tolerating a pressure of 200 to 700 psi (1.3 to 4.8 MPa).

4.1.7 Valco VICI digital valve sequence programmer for interfacing to the auto sampler.

4.1.8 Alcott 728 auto sampler

4.2 Hewlett Packard (HP) Chemstation chromatography data system equipped with HP 35900E A/D 2-channel interface.
4.3 Syringe, various sizes (1-mL to 5-mL) and equipped with a male pressure fitting.
4.4 Eluent and regenerant reservoirs, suitable containers for storing eluents and regenerant. For example, 4-L collapsible bags can be used.
4.5 Analytical balance, capable of weighing to the nearest 0.0001 g.
4.6 Weighing boats, plastic disposable to weigh standards and reagents
4.7 Pipets, Class A volumetric and Eppendorf, assorted sizes
4.8 Volumetric flasks, Class A, assorted sizes
4.9 Micro beakers, plastic disposable for sample preparation, assorted sizes
4.10 Containers for sample collection - 4-Oz HDPE bottles for water sample collection (See Section 6.1) and 4-Oz glass bottles for soil samples (See Section 6.2).
4.11 Micron filters, Gelman IC Acrodisc (0.45 µm) for filtering samples and reagents.
4.12 Conductivity meter, Fisher Scientific or Orion Model 124 equipped with a conductivity cell (e.g. Orion 012210). At a minimum, the meter should be capable of measuring matrix conductance over a range of 1 - 10,000 uS/cm.
4.13 Matrix pretreatment cartridges are used to remove sulfate, chloride, metals and other common anions such as carbonate, which may interfere with the analysis. Three separate cartridges may be used:
4.13.1 Matrix pretreatment cartridges in the barium form -- (Dionex OnGuard-Ba cartridges, PN 046072, or equivalent.) These cartridges are conditioned according to the manufacturer's directions and are used to reduce the matrix levels of sulfate.
4.13.2 Matrix pretreatment cartridges in the silver form – (Dionex OnGuard-Ag cartridges PN 039637, or equivalent.) These cartridges are conditioned according to the manufacturer's directions and are used to reduce the matrix levels of chloride.
4.13.3 Matrix pretreatment cartridges in the hydrogen form -- Dionex OnGuard-H cartridges (PN 039596) or equivalent. These cartridges are conditioned according to the manufacturer's directions and are used to reduce cations in the sample matrix. This protects the analytical column by removing silver, which has leached from the Ag cartridge and may indirectly minimize the effect of carbonate, by removing the cationic counter ion.

5.0 REAGENTS AND STANDARDS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
5.2 Reagent water. See AML SOP on Reagent water (Q-Water-Rx) for additional details. Type I water (17.8 Mohm or greater resistance) should be used for the

determination of the perchlorate or other anions. Column life may be extended by passing reagent water through a 0.22-µm filter prior to use.

5.3 Eluent solution - 25 mM sodium hydroxide (NaOH, [CASRN 1310-73-2]).

5.3.1 Dissolve 4.0 grams of 50% (W/W) sodium hydroxide in reagent water to a final volume of 2.0 L.

5.3.2 Solutions of NaOH are very susceptible to carbonate contamination resulting from adsorption of carbon dioxide from the atmosphere. This contamination will result in poor reproducibility of perchlorate retention times, elevated instrument background conductivity, and increased baseline noise/drift. Consequently, exposure to the atmosphere should be minimized by storing these eluent solutions in sealed reservoirs under low-pressure (3 to 5 psi) helium.

5.3.3 The eluent solutions should be regularly prepared and held for no more than 5 days. When refilling the eluent reservoir, completely replace old eluent solution by emptying the old eluent, rinsing the reservoir with reagent water, and refilling with the freshly prepared eluent solution.

5.3.4 With this eluent, the suppressed conductivity detector background signal should be between 2 and 5 uS. This eluent solution must be purged for 10 minutes with helium prior to use. This effectively removes dissolved gases, which may form micro bubbles in the IC, compromising system performance and adversely affecting the integrity of the data. Alternatively, an in-line degas apparatus may be employed. A system or apparatus which automatically generates the hydroxide eluent (e.g. Dionex EG40, or equivalent) is an acceptable alternative to physically preparing the hydroxide eluent.

5.4 Perchlorate Primary Standard, ACS Reagent Grade sodium perchlorate, anhydrous - Obtain from Alfa Aesar (catalogue number 11623). Prepare stock and intermediate solutions using the primary standard. Use these solutions to prepare initial calibration, continuing calibration, and spiking standard solutions.

5.4.1 Stock Primary Perchlorate Standard (1000 mg/L or 1 mg/mL) – A stock standard solution may be purchased as a certified solution or prepared from the sodium salt as listed below. (NOTE: Sodium perchlorate represents a molar weight fraction of 81.2 % perchlorate anion). Dissolve 0.1231 g sodium perchlorate (NaClO4, CASRN [7601-89-0] in reagent water and dilute to 100-mL in a volumetric flask. The stock standard expires in 12 months from the date of preparation.

5.4.2 Intermediate Primary Perchlorate Standard (1-mg/L perchlorate) - Prepare by diluting 0.50 mL of the 1000-mg/L stock solution (Section 5.4.1) to a final volume of 500 mL with reagent water. The intermediate standard expires in 12 months from the date of preparation.

5.5 Perchlorate Second source or Check Standard - Obtain from Aldrich ($NaClO_4.x\ H_2O$). Prepare check stock and intermediate solutions using the second source standard. Use these solutions to prepare initial calibration verification (ICV) standard solution.

5.5.1 Stock Second Source (Check) Perchlorate Solution (1000-mg/L perchlorate) - Dissolve 0.1231-g of the standard (or an appropriate amount

corrected for assay and water content) in reagent water and dilute to 100-mL in a volumetric flask. This second source stock standard expires in 12 months from the date of preparation.

5.5.2 Intermediate Second Source (Check) Standard (1-mg/L perchlorate) - Prepare by diluting 0.50 mL of the 1000-mg/L stock solution (Section 5.5.1) to a final volume of 500 mL with reagent water. The intermediate standard expires in 12 months from the date of preparation.

5.6 Mixed Common Anion Stock Solution - Prepare the mixed common anion stock solution containing chloride, sulfate and carbonate, each at 25 mg/mL. This solution is used to prepare simulated common anion samples in the determination of the MCT (Section 9.2.8). Dissolve the following salts in reagent water to a final volume of 25.0 mL.

5.6.1 1.0-g sodium chloride (NaCl, CASRN [7647-14-5]) = 0.61 g Cl

5.6.2 0.93-g sodium sulfate (Na2 SO4 , CASRN [7757-82-6]) = 0.63 g SO4

5.6.3 1.1-g sodium carbonate (Na2 CO3 , CASRN [497-19-8]) = 0.62 g CO3.

5.7 MCT Determination Standards - Prepare a series of solutions containing perchlorate at a fixed concentration of 25-ug/L and other common anions at 0, 200, 300, 400, 500, 600, 800, and 1000-mg/L.

5.7.1 Dilute 0.625-mL of the perchlorate intermediate primary standard (Section 5.2.2) and 0.00, 0.20, 0.30, 0.40, 0.50, 0.60, 0.80, and 1.00-mL of the mixed common anion stock solution (Section 5.5) with reagent water to a final volume of 25.0- mL into separate 25-mL volumetric flasks.

5.7.2 Label the solutions appropriately and use them for MCT determination as described in Section 9.1.

5.8 Conductivity Meter Calibration Solution - This solution contains potassium chloride (KCl, CASRN 7447-40-7, from Fisher Scientific) at a total salt weight of 745 mg/L. Dissolve 0.745-g potassium chloride in reagent water and dilute to a final volume of 1.0-L in a volumetric flask. On a properly functioning and calibrated conductivity meter, the reference conductance for this solution is 1410-uS/cm at 25°C.

5.9 INITIAL CALIBRATION STANDARDS (ICAL) - A series of primary perchlorate standard solutions used to initially establish instrument calibration and develop the calibration curve for perchlorate analysis. These are working solutions prepare from the intermediate primary standard (section 5.4.2). Prepare fresh a blank and at least five different working solutions at the following perchlorate concentrations (µg/L): 5, 10, 25, 50, and 100-µg/L.

5.9.1 Dilute 0.5-mL of the intermediate standard (Section 5.4.2) to 100-mL with reagent water in a volumetric flask to prepare the 5-µg/L standard.

5.9.2 Dilute 1.0-mL of the intermediate standard (Section 5.4.2) to 100-mL with reagent water in a volumetric flask to prepare the 10-µg/L standard.

5.9.3 Dilute 2.5-mL of the intermediate standard (Section 5.4.2) to 100-mL with reagent water in a volumetric flask to prepare the 25-µg/L standard.

5.9.4 Dilute 5.0-mL of the intermediate standard (Section 5.4.2) to 100-mL with reagent water in a volumetric flask to prepare the 50-µg/L standard.

5.9.5 Dilute 10.0-mL of the intermediate standard (Section 5.4.2) to 100-mL with reagent water in a volumetric flask to prepare the 100-µg/L standard.

5.9.6 LINEAR CALIBRATION RANGE (LCR) is defined as the concentration range over which the instrument response is linear. It is the range defined by the lowest point of calibration (5-ug/L) and the highest point (100-ug/L).

5.10 INITIAL CALIBRATION VERIFICATION STANDARD (ICV) - This is a second-source check standard solution that is analyzed immediately after a new ICAL to verify the accuracy of calibration curve for perchlorate analysis. The ICV is similar to the quality control sample (QCS) listed in EPA Method 314. This working solution is prepared fresh from the intermediate second source standard (section 5.5.2) at the following perchlorate concentration: 25-µg/L.

5.10.1 Dilute 2.5-mL of the intermediate standard (Section 5.5.2) to 100-mL with reagent water in a volumetric flask to prepare the 25-µg/L standard.

5.11 CONTINUING CALIBRATION VERIFICATION STANDARDS (CCS) - These are prepared from the primary standard solutions that are analyzed immediately before (ICCS), during (CCCS), and at the end (ECCS) of environmental sample analysis to verify instrument stability. This working solution is prepared fresh from the intermediate primary standard (section 5.4.2) at the perchlorate concentrations shown below.

5.11.1 ICCS - This 5-µg/L perchlorate standard solution is prepared at the method quantitation limit (MQL), which is the lowest point in the calibration curve. The MQL is similar to the minimum reporting level (MRL) defined in EPA Method 314. MQL is the minimum concentration that can be reported as a quantitated value for a target analyte in a sample following analysis. This defined concentration can be no lower than the concentration of the lowest calibration standard and can only be used if acceptable quality control criteria for this standard are met. This standard is analyzed daily, prior to any field sample analysis. Prepare ICCS by diluting 0.5-mL of the intermediate standard (Section 5.4.2) to 100-mL with reagent water in a volumetric flask.

5.11.2 CCCS and ECCS - This solution is prepared at the mid point of the in the calibration curve. Dilute 2.5-mL of the intermediate standard (Section 5.4.2) to 100-mL with reagent water in a volumetric flask to prepare the 25-µg/L standard.

5.11.3 CCCS - This standard is analyzed after every tenth field sample analyses, not including QC samples, which verifies the previously established calibration curve and confirms accurate analyte quantitation for the previous ten field samples analyzed.

5.11.4 ECCS - This standard is analyzed after the last field sample analyses which verifies the previously established calibration curve and confirms accurate analyte quantitation for all field samples analyzed since the last continuing calibration check (CCCS).

5.12 METHOD BLANK is equivalent to the laboratory reagent blank (LRB) sample listed in EPA Method 314. An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment,

solvents, filtration and reagents that are used with other samples. The LRB is used to determine if perchlorate or other interferences are present in the laboratory environment, the reagents, or the apparatus. The eluent or the sample diluent may also be used as MB.

5.13 Laboratory Control Samples (LCS) and LCS duplicates (LCSD) are equivalent to the laboratory fortified blank (LFM) samples listed in EPA Method 314. LCS is an aliquot of reagent water, or other blank matrix, to which a known quantity of perchlorate is added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

5.13.1 Prepare LCS and LCSD samples (25-µL perchlorate) using procedures similar to that described for the preparation of CCCS and ECCS (section 5.11.2).

5.14 Matrix Spike (MS) and MS duplicate (MSD) Samples are equivalent to laboratory fortified sample matrix (LFM) samples listed in EPA Method 314. These are aliquots of an environmental field sample to which a known quantity of perchlorate is added in the laboratory. The MS and MSD samples (LFM) are analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical result. The background concentrations of perchlorate, in the sample matrix, must be initially determined in a separate aliquot and the measured value in the MS/MSD (LFM) corrected for this background concentration. EPA Method 314 recommends that the solutions used to fortify the MS samples be prepared from the same stock used to prepare the calibration standards and not from external source stocks. This will remove the bias contributed by an externally prepared stock and focus on any potential bias introduced by the field sample matrix.

5.14.1 Prepare MS and MSD samples as described for LCS using the environmental sample instead of the eluent for dilution. Dilute 0.5-mL of the intermediate standard (Section 5.4.2) to 25-mL with the environmental sample in a volumetric flask to prepare the 25-µg/L standard. Filter the spiked sample and analyze in the same manner as the unspiked environmental sample.

5.15 LABORATORY DUPLICATE (LD) - Two sample aliquots (LD1 and LD2), taken in the laboratory from a single sample bottle, and analyzed separately with identical procedures. Analyses of LD1 and LD2 indicate precision associated specifically with the laboratory procedures by removing variation contributed from sample collection, preservation and storage procedures. The LD may be substituted with a MS/MSD pair (See Section 5.14).

5.16 PROFICIENCY TESTING (PT) or PERFORMANCE EVALUATION (PE) SAMPLE - A certified solution whose perchlorate concentration is unknown to the analyst. This sample could originate from a NELAC or other (e.g. USACE) PT sample provider. Often, results of these analyses are used as part of a laboratory certification program to objectively determine the capabilities of a laboratory to achieve high quality results. Prepare these samples according to the instructions provided with the PT sample.

5.17 METHOD DETECTION LIMIT (MDL) DETERMINATION – MDL is the minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero. Prepare a solution of perchlorate at 3 to 5 times the estimated MDL (0.5-mg/L). The recommended spiking amount is 2-µg/L. Analyze seven to nine replicates of this standard and determine MDL as described in the applicable AML SOP.

5.17.1 Prepare MDL Study Solution containing 2-µg/L perchlorate by diluting 0.2-mL of the intermediate standard (Section 5.4.2) to 100-mL with reagent water in a volumetric flask.

5.18 INSTRUMENT PERFORMANCE CHECK SOLUTION (IPC) -- A solution containing a specific concentration of perchlorate and other test substances (namely chloride, sulfate and carbonate) used to evaluate the performance of the instrument system with respect to a defined set of criteria.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

According to EPA Method 314, perchlorate has been shown to be stable for more than 28 days but extended holding time studies (beyond 35 days) were not conducted by EPA. Typically, when analytes are believed to be stable, a 28-day holding time is established as a sufficient time period to permit a laboratory to conduct the analysis. Samples may be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to insure a representative sample, allow for replicate analysis and laboratory fortified matrix analysis, if required, and minimize waste disposal. According to EPA Method 314, samples do not need to be shipped iced or stored cold in a refrigerator but every effort should be taken to protect the samples from temperature extremes. A thermally insulated sampling kit, designed to fit sampling bottles securely during shipment, should be used to protect the samples from these temperature extremes. Use the following procedures at AML.

6.1 Oblong 4-Oz HDPE bottles (Level 6 precleaned) with foamed polyethylene lined polypropylene screw cap closures (QEC cat # 6212-0004) may be used for water sample collection. Provide 1 bottle for each water sample to be collected. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples.

6.2 One 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each solid sample. Quality control samples such as MS and MSD should be treated as regular environmental samples. These samples may be shared with samples collected for other analyses such as metals and organic parameters.

6.3 Upon receipt and log-in, these sample containers should be stored in the walk-in cooler at 4°C.

6.4 Environmental water samples should be extracted and analyzed within holding times. The holding time for aqueous samples is 28 days from the day of sample collection.

7.0 HEALTH AND SAFETY

7.1 Perchlorate is a known kidney and liver toxin. However, the toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. The laboratory chemical hygiene plan should be followed to minimize exposure.

7.2 Calibration standards and other standards used for quality control may contain hazardous chemicals. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Samples may contain high levels of organic and/or inorganic contaminants, which may be hazardous. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.4 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.5 Sodium Hydroxide (NaOH), used in the preparation of the eluent is considered caustic.

7.6 Employees are responsible for safe handling of the chemicals specified in this SOP. A reference file of Material Safety Data Sheets (MSDS) is available to all personnel involved in the chemical analysis. MSDS contain written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

7.7 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8.0 POLLUTION PREVENTION, WASTE MANAGEMENT AND DISPOSAL

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities exist in laboratory operation for pollution prevention, which is the management option of first choice. When wastes cannot be feasibly reduced at the source, EPA recommends recycling as the next best option.

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal. Soil samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal. Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner.

8.3 EPA urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions.

9.0 INITIAL DEMONSTRATION OF CAPABILITY (IDC)

The EPA 314 method requires detailed setting up of the procedure in the laboratory before sample analysis could begin. Some of these procedures such as MDL determination, initial demonstration of precision, and initial demonstration of accuracy are commonly performed by the laboratory as initial demonstration of capability (IDC). However, specialized procedures such as the determination of maximum conductivity threshold require additional attention since they are specific to perchlorate analysis. Please note that procedures shown below should be followed by the analyst during IDC although some of the procedures are strictly applicable only during environmental sample analysis.

9.1 Matrix Conductivity Threshold (MCT) Determination – The MCT is an individual laboratory defined value, which should be determined by preparing a series of sequentially increasing, common anion fortified, reagent water samples each contain a constant perchlorate concentration. Initially, a reagent water prepared LCS (LFB), containing no common anions, must be analyzed which contains 25-µg/L perchlorate. Next, the series of sequentially increasing anionic solutions, each containing perchlorate at a concentration of 25 µg/L and other common anions (chloride, sulfate and carbonate), all included at uniform increasing concentrations of 200, 300, 400, 500, 600, 800, and 1000 mg/L for each anion are analyzed.

9.1.1 Perchlorate concentration of 25 µg/L can be used if the MQL is equal to or lower than 5.0- µg/L. If the laboratory's MQL is higher, choose a perchlorate concentration for MCT determination at approximately 5 times that MQL.

9.1.2 Prepare solutions containing perchlorate and common anions for MCT determination as described in Section 5.7. The solution that does not contain any of the common anions will be designated as LCS.

9.1.3 Prepare potassium chloride solution for calibration of the conductivity meter as described in Section 5.8. Calibrate the conductivity meter (Section 4.12) in accordance with procedures described in the SOP on conductance measurements.

9.1.4 Measure and record the conductance of each of these prepared solutions on a calibrated conductivity meter. The conductance values will be used to calculate MCT as described in Section 9.1.9. To use as a relative reference conductance, the 400-mg/L mixed anion sample, which contains chloride at 400-mg/L, sulfate at 400-mg/L and carbonate at 400-mg/L, should display a conductance of between 3200 uS/cm and 3700 uS/cm.

9.1.5 Calibrate the ion chromatograph for perchlorate analysis as described in Section 10.1. Analyze each MCT solution (Section 9.1.2), recording the peak area to height (A/H) ratio and the quantified concentration of perchlorate. In many data acquisition and instrument control software, the peak area to height ratio is a definable parameter which can be specified for printout on the analysis report.

9.1.6 NOTE: Both the A/H ratio and quantified perchlorate concentration for the LFB and the 200-mg/L mixed common anion solution should be reproducibly consistent but as the common anion levels increase, the A/H

ratio will also begin to increase as the peak height is distorted and reduced. As the peak is distorted, the area will also eventually begin to be distorted and the quantitated concentration will be reduced, but this is typically secondary, with the ratio of peak area to height initially predicting this pending quantitation problem.

9.1.7 Calculate the percent difference for the A/H ratio ($\%D_{A/H}$) between the average A/H ratio for the LCS (A/H_{LCS}) and the average A/H ratios for each mixed common anion solutions (A/H_{MCA}) using the following equation.

$$(\%D_{A/H}) = \frac{[(A/H_{LCS}) - (A/H_{MCA})] * 100}{(A/H_{LCS})}$$

9.1.8 NOTE: As the measured conductivity of the MCA solutions increase, the calculated $\%D_{A/H}$ for the MCA will also increase. The MCT is defined as the matrix conductance where the $\%D_{A/H}$ exceeds 20%.

9.1.9 To derive the MCT, perform a linear regression (y = m*x + c) on these data by plotting $\%D_{A/H}$ (Section 9.1.5) as the independent variable (x) versus the matrix conductance (Section 9.1.4) as the dependent variable (y). The resulting regression data should yield an r^2 value > 0.95. Record the constant or y-intercept (c) and the x-coefficient or slope (m). Calculate the MCT as follows:

$$MCT = (20 * m) + c$$

9.1.10 NOTE: An alternate procedure to the regression analysis: Establish MCT at the conductance level of the highest mixed anion solution which yielded a $\%D_{A/H}$ value below the 20 % threshold.

9.2 MQL Verification at MCT - The ability to quantitate perchlorate at the MQL in a mixed common anion solution which reflects a conductance within ±10% of that specified as the MCT should be demonstrated. This solution should contain perchlorate at the laboratory determined MQL, as well as the common anions chloride, sulfate and carbonate, prepared consistent with the instruction for the mixed anion solutions in this section and at a concentration estimated to generate a conductance near the MCT. The conductance of this solution must be measured at within ±10% of the MCT and following the analysis, the recovered perchlorate must be between 70 - 130% of the MQL concentration. If the MQL recovery fails these criteria, the MCT should be lowered by 10% and this MQL verification must be repeated.

9.3 Instrument Performance Check (IPC) at MCT - The ability to quantitate perchlorate at five times the MQL at MCT, which was determined as described in Section 9.1 or 9.2 should be verified through the analysis of an IPC. The IPC is three tiered and is used to verify the state of the IC system to quantitate perchlorate in highly ionic matrices over time. This should be conducted initially after MCT determination (AML practice) and with each analysis batch (EPA

Method 314 requirement). Please note that procedures shown below should be followed by the analyst during IDC although some of the procedures are applicable only during environmental sample analysis.

9.3.1 Prepare the IPC, a mixed common anion solution which reflects a conductance within ±10% of that specified as the MCT (Section 9.1 or 9.2, whichever is lower). This solution should contain the common anions chloride, sulfate and carbonate as well as perchlorate at a concentration of 25-µg/L, which is five times the MQL (5-µg/L).

9.3.2 Confirm the conductance of the IPC and analyze it as the initial sample in the analysis batch. If, after several weeks of storage, the measured conductance of this solution has shifted by more than 10% from the original measured value, prepare a fresh IPC solution. Following the analysis, calculate the $\%D_{A/H}$ (Section 9.1.7), by comparing the peak area to height ratio of this IPC mixed anion standard (A/H_{IPC}) for this analysis batch to the value that was derived for the LCS (A/H_{LCS}) either in the original IDC or in the previous analysis batch. As the *first tier criteria*, the value for $\%D_{A/H}$ must be less than 25% before proceeding with the analysis batch.

9.3.3 As the *second tier criteria*, the measured recovery for perchlorate in this IPC must fall between 80% and 120% (20.0-µg/L to 30.0-µg/L for a 25-µg/L fortification).

9.3.4 As the *third tier and final criteria* for the IPC, the laboratory should closely monitor the perchlorate retention time for this analysis. Small variations in retention time can be anticipated when a new solution of eluent is prepared but if sudden shifts of more than 5% are observed in the perchlorate retention time, some type of instrument problem may be present. Potential problems include improperly prepared eluent, erroneous method parameters programmed such as flow rate or some other system problem. The observed retention time for perchlorate should closely replicate the times established when the column was originally installed. As a column ages, it is normal to see a gradual shift and shortening of retention times, but if after several years of use, extensive use over less than a year, or use with harsh samples, the retention time may shift noticeably (<80% of the original recorded value). In such cases, the column may require cleaning in accordance with manufacturer's instructions or replacement. The laboratory should retain a historic record of retention times for perchlorate to provide evidence of an analytical column's continued performance.

9.3.5 If any of the conditions defined in Section 9.3.2 through 9.3.4 are not met, the MCT determination (Section 9.3.1) should be repeated and revised to a more appropriate lower matrix conductivity threshold or the problem should be identified, corrected, and IPC reanalyzed.

9.4 Initial demonstration of low system background - Laboratory Method Blank (MB) or Reagent Blank (LRB) – A MB must be prepared and treated exactly as a typical field sample including exposure to all glassware, equipment, solvents, filtration and reagents that are used with field samples. Data produced are used to

assess instrument performance of a blank sample and evaluate contamination from the laboratory environment. Values that exceed ½ the MQL indicate a laboratory or reagent contamination is present. The source of the contamination must be determined prior to conducting any sample analysis. Any sample included in an automated analysis batch which has an invalid MB, indicated by a quantitated perchlorate that exceeds ½ the MQL, should be reanalyzed in a subsequent analysis batch after the contamination problem is resolved.

9.5 Initial Demonstration of Accuracy (IDA) - Prepare and analyze 7 replicate LCS samples (Section 5.13) that have been fortified at 25.0-µg/L. Calculate the mean measured concentration (C_m) for the n replicate values (C_1, C_2, ….C_n) as shown in the following equation. To pass the IDA, the value derived for C_m should be within ± 10% of the true value or between 22.5-µg/L and 27.5-µg/L.

$$C_m = \frac{(C_1 + C_2 + C_3 + ...C_n)}{n}$$

9.6 Initial Demonstration of Precision (IDP) - Using the data generated for Section 9.5, calculate the mean (Cm as described in Section 9.5, standard deviation (std dev or S) and percent relative standard deviation (RSD) as described below or in one or more of the referenced AML SOPs. To pass the IDP, the %RSD must be less than 10%.

$$s = \sqrt{\sum_{i=1}^{n} \frac{(x_i - x)^2}{n-1}}$$

where:

s	=	standard deviation
x_i	=	measured value of the *i*th replicate
x	=	mean of the replicates (C_m)
n	=	number of replicates

$$RSD = CV = \frac{[std\ dev]}{\bar{x}}\ x\ 100$$

9.7 Initial Calibration Verification (ICV) or Quality Control Check (QCS) - After calibration curves (see Section 5.9 and …) have initially been established or have been re-established, or as required to meet data quality needs, verify both the calibration and acceptable instrument performance with the preparation and analyses of an external/second source ICV or QCS (see Section 5.10 and ….). If the determined concentrations are not within ± 10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the IDC or continuing with on-going analyses.

9.8 Method Detection Limit (MDL) – An MDL must be established using reagent water (blank) fortified at a concentration of three to five times the estimated instrument detection limit. To determine MDL values, take seven replicate aliquots of the fortified reagent water (Section) and process through the entire analytical method as described in this SOP and in applicable AML QC/QA SOPs. Perform all calculations defined in this SOP and report the concentration values in the appropriate units. Calculate the MDL as follows:

$$MDL = t_{(n-1,\ \alpha = .99)} * (s)$$

where $t_{(n-1,\ \alpha = .99)}$ is the one-sided t-statistic appropriate for the number of samples used to determine (s), at the 99 percent level. The t value is 3.14 for seven replicates, which is the default. Standard deviation (s) is calculated as shown in Section 9.6.

9.9 Method Quantitation Limit (MQL) or Minimum Reporting Level (MRL) – The MQL is the threshold concentration of an analyte that a laboratory can expect to

accurately quantitate in an unknown sample. The MQL should be established at an analyte concentration either greater than three times the MDL or at a concentration which would yield a response greater than a signal to noise ratio of five. Setting the MQL too low may cause repeated QC failure upon analysis of the ICCS. Although the lowest calibration standard may be below the MQL, the MQL must never be established at a concentration lower than the lowest calibration standard. The laboratory practice is to establish MQL at the lowest calibration standard.

10.0 CALIBRATION AND SAMPLE ANALYSIS PROCEDURES

10.1 Calibration - Demonstration and documentation of acceptable initial calibration (ICAL) is required prior to the IDC and before any samples are analyzed, and is required intermittently throughout sample analysis to meet required QC performance criteria outlined in EPA Method 314 and summarized in Table 2. Initial calibration verification (ICV) is performed using a QCS immediately after ICAL as well as with each analysis batch. For each analysis batch an initial (ICCS), continuing (CCCS) when more than 10 field samples are analyzed, and end calibration check standards (ECCS) are analyzed. The procedures for establishing the initial calibration curve (ICAL) and initial calibration verification (ICV) are described in Section 10.1.1. The procedures to verify the calibration with each analysis batch are described in Section 10.1.2.

10.1.1 INITIAL CALIBRATION CURVE (ICAL) - Establish ion chromatographic operating parameters equivalent to those indicated in Table 1. Estimate the Linear Calibration Range (LCR) to cover the expected concentration range of the field samples. The LCR should not extend over more than two orders of magnitude in concentration since it is difficult to maintain linearity throughout the entire calibration range. A minimum of three calibration standards are required for a curve that extends over a single order of magnitude and a minimum of five calibration standards are required if the curve covers two orders of magnitude. If quantification is desired over a larger range, then two separate calibration curves may be prepared or a sample dilution scheme employed. Since the anticipated concentration range for perchlorate in actual field samples is expected to cover two orders of magnitude, the use of at least five calibration standards in the range 5 to 100-µg/L is recommended.

10.1.1.1 Prepare the initial calibration standards as described in Section 5.9.

10.1.1.2 Inject 1.0 mL of each calibration standard. Tabulate peak area responses against the perchlorate concentration. NOTE: Peak areas must be used as a measure of response since they have been found to be more consistent, in terms of quantitation, than peak heights. Peak heights tend to be suppressed as a result of high levels of common anions in a given matrix which can compete for exchange sites leading to peak broadening. Using peak areas, it is

the analyst's responsibility to review all chromatograms to insure accurate baseline integration of target analyte peaks, since poorly drawn baselines will significantly influence peak areas.

10.1.1.3 The results are used to prepare a calibration curve. Acceptable calibration is confirmed after reviewing the curve for linearity (second order fits are also acceptable) and passing the criteria for the initial calibration verification (ICV or QCS) as described in Section 10.1.1.4 and initial calibration check standard (ICCS) as described in Section 10.1.2. Alternately, if the ratio of area to concentration (calibration factor) is constant over the LCR as indicated by 15% or less percent relative standard deviation (%RSD), linearity through the origin can be assumed and the average ratio or response factor can be used in place of a calibration curve. Use the instrument software to determine an appropriate calibration curve to be used for using the ICAL data.

10.1.1.4 ICV or QCS Analysis - After establishing or reestablishing calibration curves, the accuracy of this calibration must be verified through the analysis of externally prepared second source standard (ICV or QCS) as described in Section 5.10. The determined concentration for ICV must fall within ± 10% of the stated value.

10.1.2 CONTINUING CALIBRATION VERIFICATION (CCV)- Initial calibrations (Section 10.1.1) may be stable for extended periods of time. Once the calibration curve has been established it MUST be verified for each analysis batch, prior to conducting any field sample analysis using an Initial Calibration Check Standard (ICCCS). Continuing Calibration Check Standards (CCCS) and End Calibration Check Standards (ECCS) are also required as described in the sections below.

10.1.2.1 INITIAL CALIBRATION CHECK STANDARD (ICCS) – For each analysis batch the calibration must initially be verified prior to analyzing any samples. The lowest level standard used to prepare the linear calibration curve must be used. In cases where the analyst has chosen to set the MRL above the lowest standard, a standard at a concentration equal to the MRL is acceptable. Prepare ICCS standard as described in Section 5.11.1. Percent recovery for the ICCS must be in the range or 75 - 125% before continuing the analysis batch and conducting any sample analyses.

10.1.2.2 CONTINUING CALIBRATION CHECK AND END CALIBRATION CHECK STANDARDS (CCCS/ECCS) - Continuing calibration check standards should be analyzed after every tenth field sample analysis and at the end of the analysis batch as an end calibration check standard. The percent recovery for perchlorate in the CCCS/ECCS must be between 85 - 115%.

10.1.2.3 If during the analysis batch, the measured concentration for perchlorate in the CCCS or ECCS differs by more than the calibration verification criteria shown above, or if the perchlorate

peak retention time shifts outside the retention time window, all samples analyzed after the last acceptable check standard are considered invalid. These samples should be reanalyzed. The source of the problem must be identified and resolved before reanalyzing the samples or continuing analyses.

10.1.2.4 In the case where the end calibration fails to meet performance criteria, but the initial and middle calibration checks are acceptable, the samples bracketed by the acceptable calibrations may be reported. However, all field samples between the middle and end calibration checks should be reanalyzed.

10.1.3 CONDUCTIVITY METER CALIBRATION -- Prior to conducting the MCT and coinciding with each analysis batch, conductivity meter (Section 4.12) calibration must be verified or established using a standard KCl solution (Section 5.8).

10.1.3.1 Thoroughly rinse the conductivity electrode with reagent water. Place the electrode in the reagent water, turn on the meter and confirm the conductance of this blank is < 1 uS/cm.

10.1.3.2 Pour approximately 15-mL of the standard KCl solution (Section 5.8) into a plastic disposable micro beaker (Section 4.9) and place the electrode into the solution. The reference conductance for this solution is 1410 uS/cm at 25°C. The conductivity meter must yield a conductance between 1380 uS/cm and 1440 uS/cm to be in calibration.

10.1.3.3 If the conductivity meter fails calibration, recalibrate the unit per manufacture's instruction and repeat the procedure in 10.1.3.2 as if the standard solution were an unknown matrix.

10.2 Sample Preparation - Prior to conducting any field sample analysis, the conductivity of that matrix must be determined using a conductivity meter calibrated as described in Section 10.1.3. When the conductance of a field sample is above the MCT, sample dilution or pretreatment, as described in respective Sections 10.2.2 and 10.2.3 should be performed. Samples do not need to be refrigerated but if samples are held refrigerated as a standard practice for sample control, samples should be allowed to warm up to room temperature prior to conducting sample analysis. Most groundwater samples may not require any preparation other than filtering them through 0.45-µm filter into an autosampler cup.

10.2.1 MATRIX CONDUCTANCE VERIFICATION - Prior to conducting the analysis of a field sample matrix, the conductance of that matrix must be measured. Matrix conductivity is directly related to the common anion levels which, at high concentrations, can influence the integrity of the perchlorate analysis.

10.2.1.1 Verify conductivity detector calibration by following the procedure outlined in Section 10.1.3.

10.2.1.2 Pour approximately 15-mL of each sample into a plastic disposable micro beaker (Section 4.9) and reseal the sample bottle to protect it from degradation.

10.2.1.3 Place the electrode into the matrix and measure the conductivity.

10.2.1.4 If the conductance is less than the MCT, continue to Section 10.2.4.

10.2.1.5 If the conductance is greater than the MCT, the matrix requires dilution or pretreatment prior to analysis. The dilution procedure is found in Section 10.2.2. Pretreatment is described in Section 10.2.3.

10.2.1.6 Discard this aliquot of sample and be certain to thoroughly rinse the electrode with reagent water between each matrix conductivity measurement.

10.2.2 MATRIX DILUTION FOR MATRICES WHICH EXCEED THE MCT - A sample can be analyzed once diluted with reagent water to a conductance below the MCT. The exact magnitude of this dilution will adversely increase the MQL or MRL by an equivalent proportion.

10.2.2.1 Knowing the matrix conductance exceeds the MCT, estimate the proportion required for the dilution by dividing the measured matrix conductance by the MCT. Round up to the next whole number and dilute the sample by a proportion equivalent to this value. For example, if the established MCT is 6100 uS/cm and a sample reflecting a conductance of 8000 uS/cm was measured, dilute the sample with reagent water by a factor of 2.

10.2.2.2 Measure the conductance of the diluted sample to confirm it is now below the MCT. Analyze the sample as specified in Section 10.2.4 with the understanding that the MQL has now been elevated by a proportion equivalent to the dilution.

10.2.2.3 If perchlorate is measured above the elevated MQL, calculate and report actual field sample concentration taking into account sample dilution. If no perchlorate is measured above the elevated MQL and analysis or project objectives required monitoring below the concentration of the elevated MQL, proceed to Section 10.2.3 and pretreat the matrix.

10.2.3 PRETREATMENT FOR MATRICES WHICH EXCEED THE MCT – If sample dilution did not yield the required results, sample pretreatment should be employed. When the MCT is exceeded, it is most often due to high levels of common anions (chloride, sulfate, and carbonate) in a particular matrix. If the analyst were to attempt the IC analysis of this particular matrix, the common anions present in the sample would distort the baseline and negatively affect the accurate quantitation of perchlorate. To effectively reduce a significant amount of these anions that contribute to the high conductivity reading, a series of pretreatment cartridges should be employed. For this pretreatment, three cartridges are attached in series in the following order: Ba, Ag, and H. It is recommended that all three cartridges be employed unless the analyst has specific knowledge that a matrix primarily has high levels of a specific common anion.

10.2.3.1 Individually and thoroughly rinse each pretreatment cartridge with reagent water in order to insure all residual background contaminants are removed from the cartridge. Perform this rinse per manufacturer's instructions. This pretreatment is conducted by placing the cartridges in the following prescribed series (>>Ba>>Ag>>H).

10.2.3.2 Prior to pretreating any field samples, prepare and pretreat a MB (LRB) and a LCS (LFB). The LCS may be performed in duplicate. These pretreated quality control samples are required when an analysis batch contains a matrix which must be pretreated. The pretreated MB and LCS are used to verify that no background interference or bias is contributed by the pretreatment. If a response is observed in the pretreated MB, triple or quadruple the volume of reagent water rinse suggested by the manufacturer and repeat until a blank measures no more than ½ the MQL. If this additional rinsing procedure is required, it must be consistently applied to all the cartridges prior to conducting any matrix pretreatment.

10.2.3.3 Filter 3-mL of sample through the series of rinsed, stacked cartridges as an initial sample rinse at a flow rate of 1.0-mL/min or less, which is approximately one drop every 3 to 4 seconds. This flow rate is critical to the pretreatment and must be carefully followed. Discard this fraction and begin collecting the pretreated sample aliquot of collected sample.

10.2.3.4 When sufficient volume has been collected, measure the conductance of the pretreated sample aliquot being certain the conductivity meter's probe has been thoroughly rinsed and excess water has been shaken from the tip. If the conductance is now below the MCT, the sample is ready for analysis. If the conductance is still above the MCT, the flow rate through the pretreatment cartridge is likely too fast and the pretreatment should be repeated with new cartridges. In some instances, double pretreatment cartridges may need to be applied. When this pretreatment is performed properly, U.S.EPA has found 70% to 95% reduction in matrix conductance with good recoveries for perchlorate.

10.2.3.5 Prepare a MS/MSD pair for each 20 or less samples which failed the MCT and have been selected for pretreatment. Initially, the pretreated sample is analyzed and perchlorate level is determined. Then, a second and third aliquots of sample that contains perchlorate at levels less than the MS spike amount should be fortified with perchlorate, pretreated to reduce the high common anion levels, and analyzed to assess perchlorate recovery from that matrix. If the perchlorate recovery falls outside the acceptance range of 80 - 120%, that particular sample should be reported as suspect/matrix.

10.2.3.6 The pretreatment procedures prescribed above are effective at reducing the chloride and sulfate content of a sample matrix but will not reduce matrix concentrations of other anions such as nitrate or phosphate.

10.2.3.7 Proceed with sample analysis as described in Section 10.2.4.

10.2.4 FILTRATION FOR MATRICES WHICH PASS THE MCT - All samples are filtered using a 0.45-µm particulate filter prior to injection into an ion chromatograph.

10.2.4.1 Pour approximately 15-mL of sample into a micro beaker (Section 4.9) and reseal the sample bottle to protect the sample integrity.

10.2.4.2 Using a Luer lock, plastic 10-mL syringe, withdraw approximately 10-mL of sample from the micro beaker and attach a 0.45-µm particulate filter (Section 4.11), which has been demonstrated to be free of ionic contaminants, directly to the syringe.

10.2.4.3 Filter the sample into an autosampler vial or manually load the injection loop injecting a fixed amount of filtered, well mixed sample. If using a manually loaded injection loop, flush the loop thoroughly between sample analysis using sufficient volumes of each new sample matrix.

10.2.4.4 If the autosampler vials or vial caps are designed to automatically filter the sample matrix as the sample is loaded on the IC system, this filtration procedure can be omitted and the sample can be directly transferred to the autosampler vial.

10.2.5 For solid materials, the following extraction procedure should be used. Add an amount of reagent water equal to ten times the weight of solid material (weigh out to three significant numbers), e.g. 10-mL for 1-g sample. Mix the slurry for 10-minutes using a magnetic stirrer or a mechanical shaker. Allow the solid to settle down and filter the slurry using a 0.45-µm membrane type syringe filter. Analyze the aqueous extract as described for aqueous samples (Section 10.2.1 to 10.2.4).

10.3 Sample Analysis - Set up the instrument using the recommended operating conditions for the ion chromatograph which are summarized in Table 1. Perform IDC as described in Section 9. Establish a valid initial calibration and verify this calibration by analyzing a second source ICV (QCS) as described in Section 10.1.1. Analyze the IPC solution (Section 9.3), followed by the MB (LRB). Then confirm the IC system calibration by analyzing an ICCS (Section 10.1.2.1) and, if required, recalibrate as described in Section 10.1. Lastly, analyze the LCS and LCSD (LFBs). The instrument is ready for sample analysis if the QC criteria are met for the analysis described above. Follow the analytical sequence shown in Table 3.

10.3.1 Follow the example analytical sequence shown in Table 3. Analysis sequences must be carefully constructed to meet required QC specifications and frequency (Table 2). The sequence shown in Table 3 shows a hypothetical analysis batch that contains normal sample matrices (not requiring pretreatment) as well as samples which have been pretreated since they failed the MCT. Table 3 may be used as a guide when preparing analysis batches. Additional batches may be added sequentially on to the end of the sequence as long as all QC samples, which define an individual batch (IPC, MB, ICCS, LCS, LCSD, MS, MSD, etc.) are individually reanalyzed with each successive serial batch. The QC criteria for these analyses should be continually met (from the IPC through ECCS).

10.3.2 Inject 1.0-mL of each filtered sample. Use the same size loop for standards and samples. An automated constant volume injection system may also be used. Record the resulting peak size in area units and retention time for each analyte.

10.3.3 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards measured over several days. Three times the standard deviation of retention time may be used as a suggested window size but the retention time window should not extend beyond ± 5% of the retention time for perchlorate. The experience of the analyst should weigh heavily in the interpretation of these chromatograms.

10.3.4 If the response of a sample analyte exceeds the calibration range, the sample must be diluted with an appropriate amount of reagent water and reanalyzed.

10.3.5 NOTE: If dilution is not possible, then three new calibration concentrations must be employed to create a separate high concentration calibration curve, one standard near the estimated concentration and the other two bracketing around an interval equivalent to approximately ± 25% the estimated concentration. The response generated by these three new high concentration calibration standards must not exceed the upper linear range for the conductivity detector. The latter procedure involves significantly more time than a simple sample dilution therefore, it is advisable to collect sufficient sample to allow for sample dilution and sample reanalysis, if required.

10.3.6 Should more complete resolution be needed between perchlorate and a coeluting, shoulder peak, the eluent (Section 5.3) may be diluted. This will spread out the peaks, causing later elution of perchlorate.

10.3.7 NOTE: Analysts are advised to carefully evaluate any of these eluent dilutions since when these eluent changes are incorporated, other coelutions may be encountered which were not initially evident. Eluent dilution will also reduce the overall response of an anion due to chromatographic band broadening which will be evident by shortened and broadened peaks. This will adversely affect the MDLs for each analyte. The analyst should also verify that this dilution does not negatively affect QC performance.

10.3.8 AUTOMATED ANALYSIS - Large numbers of samples may need large analysis batches that are run in an automated manner. When conducting automated analyses, careful attention must be paid to ensure sufficient volume of eluent in the reservoir is available to sustain extended operation. QC performance criteria should be met throughout the analysis batch, which should be verified through subsequent careful inspection of the data.

10.4 Data Analysis and Calculations - This involves identification of the perchlorate from its retention time and calculating sample concentrations using the calibration curve.

10.4.1 Peak Identification - Identify perchlorate in the sample chromatogram by comparing the retention time of a suspect peak within the retention time window to the actual retention time of a known analyte peak in a calibration standard.

10.4.1.1 If the perchlorate retention time has slightly shifted (generally towards shorter times) since the initial calibration, but is still within acceptance criteria and are reproducible during the analysis batch, the analyst should use the retention time in the daily calibration check standards to confirm the presence or absence of perchlorate anion.

10.4.1.2 If a low concentration of perchlorate is suspected in an unknown sample, but the retention time has drifted to the edge of the retention time window, standard addition method should be employed for peak identification. A low level perchlorate MS sample containing spike amount at nearly the same concentration as the suspect peak should be prepared to confirm the matrix induced retention time shift, if any. If the fortified sample reveals a split or shouldering peak response, the low concentration in the unfortified sample is likely an interferant and should not be reported as perchlorate.

10.4.2 Compute sample concentration using the initial calibration curve generated in Section 10.1. Report results in µg/L. The HP Chemstation software can perform calibration and calculate sample results without the analyst performing any manual calculations.

10.4.3 Report those values that fall between the MQL (the lowest concentration standard on the calibration curve) and the highest calibration standards without any QC flag.

10.4.4 Report those values that fall between the MDL and the MQL as estimated with a "J" flag. This is standard SW-846 method procedure, which deviates from the EPA Method 314 guidelines. EPA Method 314 recommends samples with perchlorate identified but quantitated below the concentration established by the lowest calibration standard be reported as “trace present”.

10.4.5 Samples with a perchlorate response which exceeds the highest calibration standard concentration must be flagged as exceeding the calibration ("E" flag) and the sample reanalyzed under appropriate dilution. When this is not possible the alternate calibration procedures described in Section 10.3.5 should be followed.

10.4.6 For solid samples, multiply the mg/L results obtained by the volume of sample extract (mL) and divide by the weight (g) of the solid sample used and the percent solids.

11.0 QUALITY CONTROL

Analysis performed by the laboratory using this SOP should follow the AML quality control program. The requirements of this program consist of an initial demonstration of laboratory capability, and subsequent analysis of method required quality control (QC) samples in each analysis batch. For EPA Method 314, these requirements may include the analysis of additional QC samples: instrument performance check standard (IPC), laboratory reagent blank (MB or LRB), initial calibration check standard (ICCS), laboratory control sample (LCS) or fortified blank (LFB), continuing and end calibration check standards (CCCS/ECCS), matrix spikes (MS) or laboratory fortified sample matrix (LFM) and a field, laboratory or MS duplicate sample analysis.

11.1 Laboratory Procedure Improvements - In recognition of the rapid advances occurring in chromatography, the analyst is permitted certain options, such as the

use of different columns, injection volumes, and/or eluents, to improve the separations or lower the cost of measurements. Each time such modifications to the procedures are made, the analyst is required to repeat IDC (Section 9).

11.2 Sample Analysis - Samples which exceed the MCT must either be diluted or pretreated to reduce the common anion levels. Samples that are pretreated have additional QC requirements such as pretreated MB, LCS/LCSD, and MS/MSD. Fortification should be performed before pretreatment.

11.3 Laboratory Control Samples (LCS) - According to EPA Method 314, for normal drinking waters, the LCS typically should be prepared in the range of 20 to 50-µg/L and they should not be prepared at concentration greater than ten times the highest concentration observed in any field sample. The laboratory uses a spike level of 25-µg/L (Section 5.13). The frequency of analysis of LCS analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples. LCS can be run in duplicate to obtain precision (RPD) information. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

11.3.1 If an analysis batch only contains untreated samples, then only untreated LCS and/or LCSD samples are required.

11.3.2 When sample matrices have been pretreated to reduce the risk of high common anion interference, a second LCS must be prepared, pretreated in exactly the same manner as the pretreated samples and analyzed to confirm no background effects or recovery bias induced by the pretreatment are present.

11.3.3 If an analysis batch only contains pretreated samples, then only a pretreated LCS samples are required.

11.3.4 Analyze the LCS (filtered as if it were a field sample) with each analysis batch immediately following the ICCS.

11.3.5 Calculate accuracy as percent recovery (%R) using the equation shown in Section 11.6.4. When calculating %R for laboratory control samples (LCS), blank spikes or other reference materials, X_u could be set at zero.

11.3.6 The recovery for perchlorate should fall in the range of 85 - 115% prior to analyzing samples.

11.3.7 If the LCS recovery for an analysis batch does not meet these recovery criteria the data are considered invalid, and the source of the problem should be identified and resolved before continuing analyses.

11.4 Laboratory Method Blank (MB) or Reagent Blank (LRB) – A MB must be prepared and treated exactly as a typical field sample including exposure to all glassware, equipment, solvents, filtration and reagents that are used with field samples. Data produced are used to assess instrument performance of a blank sample and evaluate contamination from the laboratory environment.

11.4.1 The frequency of MB analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples.

11.4.2 When sample matrices have been pretreated to reduce the risk of high common anion interference, a second MB must be prepared, pretreated in

exactly the same manner, and analyzed to confirm no background effects from the pretreatment process are present. If an analysis batch only contains pretreated samples, then only a pretreated MB is required.

11.4.3 Values that exceed ½ the MQL indicate a laboratory or reagent contamination is present. The source of the contamination must be determined prior to conducting any sample analysis.

11.4.4 Any sample included in an automated analysis batch which has an invalid MB, indicated by a quantitated perchlorate that exceeds ½ the MQL, should be reanalyzed in a subsequent analysis batch after the contamination problem is resolved.

11.5 Matrix Effects - Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on knowledge of the samples in the sample batch. If samples are not expected to contain target analytes, the laboratory should use a matrix spike and matrix spike duplicate pair, spiked with the anion at the mid-point of the initial calibration curve. If samples are expected to contain the target analyte, then the laboratory may use one matrix spike and a duplicate analysis of an unspiked field sample. Since this knowledge may not always be available, AML will always analyze the MS in duplicate (MS and MSD). Sample duplicate analyses need not be performed unless client requests duplicate analysis for a specific project. Duplicate analyses should be performed if the sample selected for MS contains large amounts of the target analyte(s).

11.6 Matrix spikes (MS) or laboratory fortified matrix (LFM) - MS and MS duplicate (MSD) samples are equivalent to laboratory fortified sample matrix (LFM) samples listed in EPA Method 314. These are aliquots of an environmental field sample to which a known quantity of perchlorate is added in the laboratory. The MS and MSD samples (LFM) are analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical result. The background concentrations of perchlorate, in the sample matrix, must be initially determined in a separate aliquot and the measured value in the MS/MSD (LFM) corrected for this background concentration.

11.6.1 The frequency of analysis of MS analysis is equivalent to a minimum of 2 per 20 samples or 2 per batch if less than 20 samples. MS is run in duplicate (MS and MSD) to obtain precision (RPD) information.

11.6.2 Samples that exceed the MCT must either be diluted or pretreated to reduce the common anion levels. Samples that are pretreated should be accompanied by a separate set of MS/MSD samples that should be fortified before pretreatment.

11.6.3 The fortified concentration must be equal to or greater than the native sample concentration. Fortified samples that exceed the calibration range must be diluted to be within the linear range. In the event that the fortified level is less than the observed native level of the unfortified matrix, the recovery should not be calculated. This is due to the difficulty in

calculating accurate recoveries of the fortified concentration when the native sample concentration to fortified concentration ratio is greater than one.

11.6.4 Calculate the percent recovery (%R) for each target analyte, corrected for concentrations measured in the unfortified sample. Percent recovery should be calculated using the equations shown below:

$$\%R = \frac{(x_s - x_u)}{K} x\,100$$

where:

X_s = measured value of the spiked sample

X_u = measured value of the unspiked sample

K = Amount spiked to the sample

11.6.5 Recoveries may exhibit matrix dependence. If the recovery for perchlorate falls outside 80 - 120% for aqueous samples (70 - 130% for soil samples), and the laboratory's performance for all other QC performance criteria is acceptable, the accuracy problem encountered with the fortified sample is judged to be matrix related, not system related.

11.6.6 The result in the unfortified sample and the MS/MSD (LFM) can be labeled as suspect/matrix to inform the data user that the result is suspect due to matrix effects. Repeated failure to meet recovery criteria may indicate potential problems with the procedure and should be investigated.

11.7 FIELD, LABORATORY DUPLICATES OR DUPLICATE LFM (MSD) – See Section 11.5 for guidance on this topic. The laboratory should analyze a field duplicate, a laboratory duplicate, or a duplicate LFM (MSD) for a minimum of 5% of the collected field samples or at least one with every analysis batch, whichever is greater. The sample matrix selected for this duplicate analysis must contain measurable concentrations of the target anions in order to establish the precision of the analysis set.

11.7.1 Calculate the relative percent difference (RPD) of the initial quantitated concentration (x_1) and the duplicate quantitated concentration (x_2) using the equation shown below:

$$RPD = \frac{|x_1 - x_2|}{(x_1 + x_2)/2} x\,100$$

11.7.2 Duplicate analysis may exhibit matrix dependence. If the RPD for the duplicate measurements of perchlorate falls outside ± 15% for aqueous samples (± 30% for soil samples) and if all other QC

performance criteria are met, laboratory precision is out of control for the sample and perhaps the analytical batch.

11.7.3 The result for the sample and duplicate should be flagged to inform the data user that the result is suspect due to a potential matrix effect, which led to poor precision.

11.7.4 This should not be a chronic problem and if it frequently recurs (>20% of duplicate analyses), it indicates a problem with the instrument or individual technique that should be corrected.

11.8 Instrument Calibration - Perform initial calibration (ICAL), initial calibration verification (ICV) with a second source standard, and continuing calibration verification (ICCS, CCCS, and ECCS) as described in Section 10.1.1 and 10.1.2.

11.9 Instrument Performance Check - Analyze IPC samples with each batch of environmental samples as described in Section 9.3.

11.10 External proficiency evaluation or testing (PE or PT) samples - The laboratory should perform analysis of quality control check samples and participate in relevant proficiency testing (PT) or performance evaluation (PE) sample studies. The PE or PT samples may be available through USACE or NELAC vendors.

12.0 **DATA VERIFICATION AND VALIDATION -** This section will provide guidance when evaluating and validating data generated by this SOP. It provides procedures to assess if data produced is valid and legally defensible. Document the review on the checklist (See Appendix 6 for details) and on the case narrative as appropriate. A number of appendices that contain chromatograms, example reports, checklists, Excel spreadsheets, etc. are referenced in this document. The list of appendices is shown in Table 6.

12.1 HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

12.1.1 WATER - The holding time for perchlorate is 28-days. Holding times are calculated in days from the date of sampling. Holding times are established by comparing the information in the chain of custody/sample log-in information with the date of sample injection.

12.1.2 SOIL / SOLIDS– A 14 day holding is used for soil and waste samples for the preparation of sample extracts. Aqueous holding times will apply to the sample extracts. The holding times for sample extraction is calculated in days from the date of sampling.

12.1.3 ACTION – If holding times have been exceeded, contact the client. All positive results may require qualification as estimated (J) or unusable (R) or invalid (I) by the user (client). Include a statement in the case narrative indicating holding time outliers. If the holding times are greatly exceeded, then the reviewer (client)

may use professional judgement to invalidate sample results as unusable.

12.2 INSTRUMENT PERFORMANCE CHECK (IPC) - The analysis of IPC standard is a three tiered system, which is used to verify the state of the ion chromatography system over time to quantitate perchlorate in highly ionic matrices. See Section 9.3 for more details. Document IPC results on laboratory notebooks (See Appendix 2 for details).

12.2.1 The conductance of the IPC solution should be within ±10% of the MCT.
12.2.2 ACTION - Prepare a new IPC solution.
12.2.3 The observed $\%D_{A/H}$ for the IPC should be less <25%.
12.2.4 ACTION - Correct chromatography problems, revise MCT if necessary and reanalyze IPC.
12.2.5 The observed perchlorate value for the IPC standard should be within ±20% of the true value.
12.2.6 ACTION – Correct the problem and reanalyze IPC.
12.2.7 The retention time for perchlorate should be within 80% of the value determined during IDC.
12.2.8 ACTION - Determine the source of the problem (column degradation, etc.) and correct it. Repeat MCT determination, perform a new ICAL and reanalyze for an acceptable IPC.

12.3 INITIAL CALIBRATION – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the IC is capable of producing data within a range that is linear. Document ICAL results in Form-1 equivalents (See Appendix 1 for details).

12.3.1 Calibration Factor Method – The %RSD should be less than 15%.
12.3.2 ACTION – Use this procedure to calculate sample results or use linear regression.
12.3.3 Linear Regression - The correlation coefficient should be greater than 0.995 over the LCR.
12.3.4 ACTION – Use this procedure to calculate sample results, drop the high point and recalculate correlation coefficient or use nonlinear regression.
12.3.5 Nonlinear Regression - Use a second order fit and calculate the weighted coefficient of determination (COD) as described in SW-844 method 8000B, Section 7.5.3. The COD should be equal to or greater than 0.99. Nonlinear regression is generally not encouraged.
12.3.6 ICV - The observed value for the second source standard should be within ±10%.

12.3.7 ACTION - correct instrumental problem, if any and reanalyze ICV. If the problem is not solved, replace the second source standard and repeat ICV. Prepare a fresh set of ICAL solutions and repeat the initial calibration procedure.

12.3.8 ACTION - If the situation cannot be corrected within the holding times of the samples, perform sample analysis using a narrow range of calibration. Positive results for compounds not meeting the criteria may need to be qualified as estimated (J) by the user (client). Report detects and non-detects without qualifiers and mention these outliners in the case narrative.

12.4 CONTINUING CALIBRATION VERIFICATION – The continuing calibration verification is used to check that the instrument is still operating under the linear range of the initial calibration and has remained stable. See Section 10.1.2 for more details. Document CALV (ICV and CCV) results in Form-7 equivalents (See Appendix 2 for details).

12.4.1 ICCS – The observed value for the ICCS standard should be within ±25% of the true values.

12.4.2 ACTION – Correct the problem and reanalyze ICCS.

12.4.3 CCCS and/or ECCS - The observed value for these standards should be within ±15% of the true values.

12.4.4 ACTION – Correct the problem and reanalyze CCCS/ECCS and all associated samples.

12.5 METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Sample data qualification to alert clients about blank contamination is performed by the laboratory. Sample data qualification based on all available blank results (laboratory MB, equipment blank, and other field blanks) is performed by the user (client). The processing of blanks helps eliminate reporting of false positives in project reports.

12.5.1 Method blanks should be free of all target anaytes and other interfering non-target analytes for the treated and untreated samples. To be acceptable, blank results should not exceed one half of the applicable MQL.

12.5.2 Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples. If levels greater than one half the MQLs are present, inspect and correct the problem. Reanalyze all associated samples. Contact client if other criteria are applicable. The only exception may be quick turn around projects.

12.5.3 ACTION – All positive results are reported with a "B" code, if contaminants are found in the method blank.

12.5.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, then no action is taken.

12.5.5 ACTION – Any compound detected in the sample which were also detected in associated method blank at levels greater than one half the MQL should be re-extracted and/or re-analyzed unless other client criteria are applicable.

12.6 LABORATORY CONTROL STANDARD (LCS) – A LCS analysis is used to evaluate method competence in the absence of matrix effects. The LCS may provide a mechanism to check method efficiency for target compounds without the sample matrix as a factor. Document LCS/LCSD results in Form-1 equivalents and recoveries/RPD in Form-3 equivalents (See Appendix 3 for details).

12.6.1 The LCS recoveries should fall in the 85-115% (±15% of the true values) range for the treated and untreated samples. Flag outliers with an asterisk (*).

12.6.2 ACTION – Correct the problem and reanalyze all associated samples.

12.6.3 ACTION - If LCS is analyzed in duplicate and one of the recoveries for one of the LCS standards is within QC limits, reanalysis of the associated samples need not be performed if chromatographic anomalies are observed only for the failed LCS.

12.7 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike data are used to evaluate the precision and accuracy of the analytical method on project samples. Document MS/MSD results in Form-1 equivalents and recoveries/RPD in Form-3 equivalents (See Appendix 4 for details).

12.7.1 The MS and MSD recoveries should fall in the 80-120% (±20% of the true values) range for the treated and untreated aqueous samples. Use ±20% of the true value criteria for soil samples. Flag outliers with an asterisk (*).

12.7.2 ACTION – Perform one manual calculation using raw data and extraction sheet information. Reintegrate spiked and/or unspiked samples if chromatographic anomalies are observed and recalculate results.

12.7.3 ACTION - No action is taken on MS/MSD recoveries data alone to qualify associated samples. Since the presence of significant amounts of target and/or non-target anions and matrix interference could lead to lead to serious analytical problems, the supervisor should be consulted when significant outliers are noted. Document QC outliers on the case narrative. See Section 11.6 for additional guidance.

12.8 DUPLICATES – Calculate RPD between duplicate results. Duplicate results may be obtained from the determination of LCS/LCSD results, MS/MSD results, laboratory duplicate sample analysis, and field sample duplicate analysis if the field duplicates are not blind. Use only sample results. Recoveries should not be used to calculate RPDS. RPD is a measure of the precision. Document RPDs in Form-3 equivalents as described for LCS and MS duplicates (See Appendix 3 and 4 for details).

12.8.1 The RPD should be <15% for aqueous samples (<30% for soil). Flag outliers with an asterisk (*).

12.8.2 ACTION – Document QC outliers for RPD on the checklist and on client reports (*) and draw attention to them on the case narrative. No action may be taken on RPD alone either to reanalyze samples and/or to qualify associated samples by the analyst. The affected sample may be qualified by the end user as having matrix-related problems. Professional judgment may be used by the user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

12.9 TARGET ANION IDENTIFICATION – Criteria for qualitative analysis is to minimize the number of erroneous positive or negative identification of the anions. Criteria for quantitative analysis includes specifications for calibration and associated quality control samples as described in the previous sections. Client specifications (e.g. USACE, AFCEE, etc.) for method performance should also be considered. Contact Supervisor (See also Appendix 10 for details) for project information. Document sample results in Form-1 equivalents (See Appendix 5 for details) and retention time (RT) windows (See Appendix 7 for RT window studies determination). Use MDLs, and MQLs (See Tables 4 and 5) and MCT information documented in the logbooks (See also Appendix 8 and 9 for details).

12.9.1 Sample conductance should not exceed MCT.

12.9.2 ACTION - Analyze sample under dilution or after pretreatment. Separate QC sample sets (MB, LCS/LCSD, MS/MSD) are needed for the treated and untreated samples.

12.9.3 The retention time (RT) for positive identification of anions in samples should be within the RT windows established for each anion.

12.9.4 ACTION – Professional judgement must be used when applying the RT window criteria since RT shifts are quite common due to significant difference between the sample and calibration media.

12.9.5 Report positive results that are greater than either the MDL and/or the reporting limit (as appropriate) but lower than the MQL as estimated with a "J" flag. Report results greater than the upper

limit of calibration with as estimated with an "E" flag. Such samples will require reanalysis with dilution. Adjust sample results and associated MDL and MQL, for percent moisture and non-standard sample weights. See Section 10.3 for additional guidance.

13.0 REFERENCES

13.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9058, Determination of Perchlorate using Ion Chromatography with Chemical Suppression Conductivity Detection, Revision 0, November 2000.

13.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9056, Determination of Inorganic Anions by Ion Chromatography, September 1994.

13.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.

13.4 Environmental Protection Agency. Test Method for the Determination of Inorganic Anions in Water by Ion Chromatography, Method 300.0. EPA-600/4-84-017, Revised document dated December 1989.

13.5 Test Method for the Determination of Inorganic Anions in Water by Ion Chromatography, U.S. EPA Method 300.1. EPA Document number: EPA/600/R-98/118. NTIS number PB98-169196 INZ.

13.6 Annual Book of ASTM Standards, Volume 11.01 Water D4327, Standard Test Method for Anions in Water by Ion Chromatography, pp. 696-703. 1988.

13.7 Standard Methods for the Examination of Water and Wastewater, Method 4110B, "Determination of Anions by Ion Chromatography-Ion Chromatography with Chemical Suppression of Eluent Conductivity," 19th Edition, 1995.

13.8 Dionex, Operation and Maintenance Manual, Dionex Corp., Sunnyvale, CA 94086.

13.9 Analysis of Low Concentrations of Perchlorate in Drinking Water and Ground Water by Ion Chromatography, Application Note 121, Dionex Corporation, Sunnyvale, CA (1998).

13.10 Anion Self-Regenerating Suppressor (ASRS) Quickstart Procedure, Document Number 031368-01, Dionex Corporation, Sunnyvale, CA, March,1988.

13.11 Installation Instructions and Troubleshooting Guide for the Anion Self-RegeneratingSuppressor-Ultra, Document Number 031367, Rev. 03, Section 5.1, Dionex Corporation, Sunnyvale, CA, December, 1988.

13.12 Method detection limit (MDL) as described in "Trace Analyses for Wastewater," J. Glaser, D. Foerst, G. McKee, S. Quave, W. Budde, Environmental Science and Technology, Vol. 15, Number 12, p. 1426, December 1981.
13.13 Gaskill, A.; Estes, E. D.; Hardison, D. L.; and Myers, L. E. Validation of Methods for Determining Chlorine in Used Oils and Oil Fuels. Prepared for U.S. Environmental Protection Agency Office of Solid Waste. EPA Contract No. 68-01-7075, WA 80. July 1988.
13.14 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.
13.15 AML Standard Operating Procedures, Q-Water-RC.
13.16 AML Standard Operating Procedures, Q-WasteMgmt-RD.
13.17 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
13.18 AML Standard Operating Procedures, Q-ControlCharts-RC.
13.19 AML Standard Operating Procedures, Q-Training-RE.

TABLE 1

Analytical Parameters for Perchlorate Analysis

PARAMETER	AML RECOMMENDATION
Ion Chromatograph	Dionex DX-120
Sample Loop	1000-µL
Eluent	25 mM NaOH
Eluent Flow	1.5 mL/Min.
Column	Dionex AS16, 4-mm
Typical back pressure	1700 psi
Temperature	Ambient
Suppressor	ASRS ULTRA, 300 MA current
Detector	Suppressed Conductivity Detector, Dionex Background conductivity: 2 to 3 µS
MCT	µS/cm
Perchlorate Retention Time	20 min.
Analysis Time	25 min.
MDL Spike Level	2-µg/L perchlorate
LCS Spike Level	25-µg/L perchlorate
MS Spike Level	25-µg/L perchlorate
ICAL Standard-1	5-µg/L perchlorate
ICAL Standard-2	10-µg/L perchlorate
ICAL Standard-3	25-µg/L perchlorate
ICAL Standard-4	50-µg/L perchlorate
ICAL Standard-5	100-µg/L perchlorate
Linear Calibration Range (LCR)	5 to 100-µg/L perchlorate
ICV Standard	25-µg/L perchlorate
ICCS Standard	5-µg/L perchlorate
CCCS Standard	25-µg/L perchlorate
ECCS Standard	25-µg/L perchlorate
IPC Standard	25-µg/L perchlorate

TABLE 2-A
QC Performance Criteria from EPA Method 314
Initial Demonstration of Capability

QC Acronym	QC Description	Specification & Frequency	Acceptance Criteria
IDC	Initial Demonstration of Capability	Prior to Sample Analysis	See Below
IDLB	Initial Demonstration of Low System Background (Method Blank)	Prior to performing analysis for IDC	<1/2 of MQL (2.5-µg/L)
IDA	Initial Demonstration of Accuracy	Seven replicates spiked at 25-µg/L - Determine Percent Recovery	Mean within ±10% of true value (22.5 to 27.5-µg/L)
IDP	Initial Demonstration of Precision	Seven replicates spiked at 25-µg/L - Determine Percent Relative Standard Deviation	%RSD ≤10%
ICAL	Initial Calibration	5-point initial calibration before IDA, IDP and MDL determination	%RSD for CF≤15% or Correlation coefficient for LR ≥0.995
ICV or QCS	Quality Control Check Sample	Immediately after ICAL - Second source ICV Standard at 25-µg/L	ICV Value within ±10% of true value (22.5 to 27.5-µg/L)
MDL	Method Detection Limit	Seven replicates spiked at 2-µg/L - Determine Standard Deviation (s)	MDL = 3.14*(s) Mean found ≤ 5*MDL
MQL or MRL	Method Quantitation Limit	Lowest calibration Point on the ICAL (5-µg/L)	MQL ≥ low ICAL standard
MCT	Matrix Conductivity Threshold	Seven solutions spiked at 25-µg/L and increasing levels of common anions	$\%D_{A/H} \leq 20\%$
MQLV	MQL Verification	5-µg/L standard containing common anions at MCT	Value within ±30% of true value (3.5 to 6.5-µg/L)
IPC	Instrument Performance Check	25-µg/L standard containing common anions at MCT	Value within ±20% of true value (20 to 30-µg/L)

TABLE 2-B
QC Performance Criteria from EPA Method 314
Calibration and Quality Control for Each Analysis Batch

QC Acronym	QC Description	Specification & Frequency	Acceptance Criteria
ICAL and ICV	Initial Calibration	An acceptable and verifiable 5-point ICAL before sample analysis ICV (25-µg/L)	%RSD for CF≤15% Corr. Coeff. For LR ≥0.995, ICV result within ±10% of TV
IPC	Instrument Performance Check	Conductance: $\%D_{A/H}$: Perchlorate Found: Retention Time: Analyze IPC First	Within ±10% of MCT <25% ±20% of TV <5% shift from IDC
MB or LRB	Method Blank or Lab reagent Blank	Prior to analyzing ICCS With each analytical batch of <20 samples	<1/2 of MQL (2.5-µg/L) for perchlorate
ICCS	Initial Calibration Check Standard (MQLV)	MQL verification at MCT - immediately after LRB analysis	Perchlorate Found should be ±25% of True Value
LCS and LCSD	Laboratory Control Samples or Laboratory Fortified Blanks (LFB)	Prior to analyzing each analytical batch of <20 samples	Perchlorate Found should be ±15% of True Value RPD ≤ 20%
CCCS	Continuing Calibration Check Standard	Mid-level (25-µg/L) standard after every 10 samples	Perchlorate Found should be ±15% of True Value
ECCS	Ending Calibration Check Standard	Mid-level (25-µg/L) standard after the last sample in batch	Perchlorate Found should be ±15% of True Value
MS and MSD	Matrix Spike Samples or Laboratory Fortified Matrix (LFM)	With each analytical batch of <20 samples	Perchlorate Found should be ±20% of True Value RPD ≤ 20%
Pretreated MB or LRB	Method Blank or Lab reagent Blank for Pretreated Samples	With each analytical batch of <20 pretreated samples	<1/2 of MQL (2.5-µg/L) for perchlorate
Pretreated LCS and LCSD	Laboratory Control Samples or Laboratory Fortified Blanks (LFB) for pretreated samples	With each analytical batch of <20 pretreated samples	Perchlorate Found should be ±15% of True Value RPD ≤ 20%
Pretreated MS and MSD	Matrix Spike Samples or Laboratory Fortified Matrix (LFM) for pretreated samples	With each analytical batch of <20 pretreated samples	Perchlorate Found should be ±20% of True Value RPD ≤ 20%

TABLE 3
Analytical Sequence

Injection #	Sample or Standard description
1	ICAL-Blank
2	ICAL-Standard-1 (5-µg/L)
3	ICAL-Standard-2 (10-µg/L)
4	ICAL-Standard-3 (25-µg/L)
5	ICAL-Standard-4 (50-µg/L)
6	ICAL-Standard-5 (100-µg/L)
7	ICV (25-µg/L second source)
8	IPC (25-µg/L at MCT)
9	MB (Untreated Samples)
10	ICCS (5-µg/L MQL Standard at MCT)
11	LCS (Untreated Samples)
12	LCSD (Untreated samples)
13	Sample 1
14	Sample 1-MS (Untreated Sample)
15	Sample 1-MSD (Untreated Sample)
16	Sample 2
17	Sample 3
18	Sample 4
19	Sample 5
20	Sample 6
21	Sample 7
22	Sample 8
23	CCCS (25-µg/L standard)
24	Sample 9
25	Sample 10
26	Sample 11
27	Sample 12
28	Pretreated-MB
29	Pretreated-LCS
30	Pretreated-LCSD
31	Pretreated Sample 12-
32	Pretreated Sample 12-MS
33	Pretreated Sample 12-MSD
34	Pretreated Sample 13
35	Pretreated Sample 14
36	CCCS (25-µg/L Standard)
37	Sample 15
38	Sample 16
39	Sample 17
40	Sample 18
41	Sample 19
42	Sample 20
43	ECCS (25-µg/L Standard)

TABLE 4
Aqueous MDLs, MQLs, and QC Limits for LCS, MS/MSD Recoveries and RPD

Perchlorate Analysis by Ion Chromatography

Analytical Management Laboratories

Matrix:	**Water**		Preparation Method:			EPA-314.0/SW-9058				MDL Dates:			11/01 to 4/02			
			Analytical Method:			EPA-314.0/SW-9058				IDP Dates:			11/01 to 4/02			
	Study	Sample	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**		**LCS Limits**		**MS Limits**		**RPD**
Analyte	Date	Loop	Amt.	ug/L	ug/L	**Ug/L**	RD=	flag	**ug/L**	RQ=	flag	Lower	Upper	Lower	Upper	Limit
		(uL)	ug/L	B		**A**	B/A		**C**	C/A		**%R**	**%R**	**%R**	**%R**	%
Perchlorate	11/16/01	180	50.0	59.0	6.9	**21.8**	2.7		100	4.6		85	115	80	120	15
Perchlorate	4/16/02	250	5.0			**2**			5			85	115	80	120	15
Mean Ratio (RD or RQ):							2.7			4.6						

MDL= ST Dev * t factor (3.14 for 7 reps)

MDLs and MQLs are subject to change due to ongoing method implementation changes.

Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 5.

Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.

LCS and MS %R limits are laboratory limits with guidance from USACE EM200-1-3, Appendix I.

TABLE 5
Soil MDLs, MQLs, and QC Limits for LCS, MS/MSD Recoveries and RPD

Perchlorate Analysis by Ion Chromatography

Analytical Management Laboratories

Matrix:	**Soil**		Preparation Method:			EPA-314.0/SW-9058				MDL Dates:			11/01 to 4/02			
			Analytical Method:			EPA-314.0/SW-9058				IDP Dates:			11/01 to 4/02			
	Study	Sample	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**		**LCS Limits**		**MS Limits**		**RPD**
Analyte	Date	Loop	Amount	ug/kg	ug/kg	**ug/kg**	RD=	flag	**ug/kg**	RQ=	flag	Lower	Upper	Lower	Upper	Limit
		(uL)	ug/kg	B		**A**	B/A		**C**	C/A		**%R**	**%R**	**%R**	**%R**	%
Perchlorate	11/16/01	180	500	590	69	**220**	2.7		100	0.5	*	85	115	70	130	30
Perchlorate	4/16/02	250	50			**20**			50			85	115	70	130	30
Mean Ratio (RD or RQ):							2.7			0.5						

MDL= ST Dev * t factor (3.14 for 7 reps) - Soil MDLs are estimated based on water MDLs and dilution factor (10).

MDLs and MQLs are subject to change due to ongoing method implementation changes.

Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 10.

Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.

LCS and MS %R limits are laboratory limits with guidance from USACE EM200-1-3, Appendix I.

TABLE 6
List of Appendices

Appendix Number	Appendix Topic
1	Initial Calibration (ICAL) Summary (Form-6 Equivalents) Initial Calibration Verification (ICV) Summary (Form-7 Equivalents) Instrument Printouts
2	Instrument Performance Check (IPC) Summary Calibration Verification (ICCS, CCCS, ECCS) Summary (Form-7 Equivalents) Instrument Printouts
3	Laboratory Control Sample (LCS or LFB) Results (Form-1 Equivalents) LCS/LCSD Recoveries and RPD Summary (Form-3 Equivalents) Instrument Printouts
4	Matrix Spike (MS/MSD or LFM) Results (Form-1 Equivalents) MS/MSD Recoveries and RPD Summary (Form-3 Equivalents) Instrument Printouts
5	Sample Results (Form-1 Equivalents)
6	Checklist for Data Review and Verification
7	Retention Time Window Studies Summary
8	Method Detection Limit (MDL) Studies Summary
9	Maximum Conductivity Threshold (MCT) MCT Summary
10	Specifications for Perchlorate Analysis by Ion Chromatography (EPA 314.0, SW-9058, AFCEE, USACE, etc.)

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. I-9056GN-RD

DETERMINATION OF GUANIDINE NITRATE BY ION CHROMATOGRAPHY (EPA 300.0 and SW-846 9056)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Analytical Management Laboratories

DETERMINATION OF GUANIDINE NITRATE BY ION CHROMATOGRAPHY (EPA 300.0 and SW-846 9056)

1.0 SCOPE AND APPLICATION

This Analytical Management Laboratories (AML) standard operating procedure (SOP) for the analysis of guanidine nitrate (GN) is based on Alliant Techsystems operating procedure HD-5-3-7210, SW-846 Method 9056, EPA 300.0, and instrument manufacturer's recommended procedures.

1.1 This method addresses the determination of guanidine nitrate in aqueous (groundwater, surface water, drinking water, wastewater, sumps, lagoons, liquid waste treatment (LWT), etc.) and soil samples.

1.2 The method detection limit (MDL) for GN is approximately 0.2-mg/L guanidinium ion in aqueous matrices. Guanidine nitrate is reported as the guanidinium ion.

2.0 SUMMARY OF METHOD

2.1 A small volume of the aqueous sample or the soil extract solution, typically 0.1-mL, is injected into an ion chromatograph to flush and fill a constant volume sample loop. The sample is then injected into a stream of eluent of the same strength as the water sample or extract.

2.2 The sample is pumped through three different ion exchange columns and into a conductivity detector. The first two columns, a precolumn or guard column and a separator column, are packed with cation exchangers. Ions are separated into discrete bands based on their affinity for the exchange sites of the resin. The last column is a suppressor column that reduces the background conductivity of the eluent to a low or negligible level. The separated cations are measured using an electrical-conductivity cell. Analytes are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a calibration curve generated from known standards.

3.0 INTERFERENCES

3.1 Any species with retention times similar to that of GN will interfere. Large quantities of ions eluting close to the ion of interest will also result in interference. Separation can be improved by adjusting the eluent concentration and/or flow rate. Sample dilution and/or the use of the method of standard additions can also be used. For example, high levels of other cations such as ammonium, K, Ca, Fe, Mg, and Na, minerals, and organic compounds that may be present in samples may interfere with the analysis.

3.2 GN samples do not require preservation with chemical additives. Samples preserved with acid(s) may cause severe interference.

3.3 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

3.4 Samples that contain particles larger than 0.45 µm and reagent solutions that contain particles larger than 0.20 µm require filtration to prevent damage to instrument columns and flow systems.

4.0 APPARATUS AND MATERIALS

4.1 Ion chromatograph, Dionex DX-120, capable of delivering 1 to 5 ml of eluent per minute at a pressure of 1100 to 1500 psi. The chromatograph is equipped with an injection valve, a 25- to 1000-µL sample loop, and set up with the following components.

4.1.1 Cation precolumn, a guard column placed before the separator column to protect the separator column from being fouled by particulates or certain organic constituents (Dionex IonPac CG-12, P/N 44002).

4.1.2 Cation separator column, a column packed with low-capacity pellicular cation exchange resin that is suitable for resolving GN from other cations (Dionex IonPac CS-12, P/N 44001).

4.1.3 Cation self-regenerating suppressor column, a column that is capable of converting the eluent and separated cations (Dionex CSRS, P/N 46079).

4.1.4 Detector, a low-volume, follow-through, temperature-compensated, electrical conductivity cell (approximately 6 µL volume, Dionex CDM-3 standard cell) equipped with a meter capable of reading from 0 to 1,000 µseconds/cm on a linear scale.

4.1.5 Pump, capable of delivering a constant flow of approximately 2 to 5 ml/min throughout the test and tolerating a pressure of 200 to 700 psi (1.3 to 4.8 MPa).

4.1.6 Valco VICI digital valve sequence programmer for interfacing to the auto sampler.

4.1.7 Alcott 728 auto sampler

4.2 Hewlett Packard (HP) Chemstation chromatography data system equipped with HP 35900E A/D 2-channel interface.

4.3 Syringe, minimum capacity of 1 ml and equipped with a male pressure fitting.

4.4 Eluent and regenerant reservoirs, suitable containers for storing eluent, flush solution, cleaner solutions, and regenerant. Carboys and 4-L collapsible bags can be used.

4.5 Analytical balance, capable of weighing to the nearest 0.0001 g.

4.6 Weighing boats

4.7 Pipets, Class A volumetric flasks and 1000-uL Eppendorf
4.8 Beakers, assorted sizes
4.9 Volumetric flasks, Class A assorted sizes
4.10 Compressed gases, helium and air
4.11 Filters, 25-mm 0.45 micron syringe filters
4.12 Graduated cylinders, 1-L
4.13 Sample vials to fit Alcott 728 autosampler
4.14 Carboys, assorted sizes (1-L, 20-L, etc.)
4.15 Collapsible bags, various size

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Reagent water. See AML SOP on Reagent water (Q-Water-Rx) for additional details. Type I water should be used for the determination of the anions. Column life may be extended by passing reagent water through a 0.22-µm filter prior to use.

5.3 Guanidine Nitrate, Aldrich Chemical, catalogue number, 23,424-9

5.4 Guanidine Nitrate, second-source, ACROS Organics, catalogue number, 21531-1000

5.5 Methanesufonic acid (MSA), anhydrous, 99.5% purity, Aldrich Chemical, catalogue number, 47,135-6. Use this neat chemical to prepare eluent and cleaning solutions as described below. NOTE: MSA is a corrosive toxic liquid. Handle with care.

5.5.1 Cation Eluent (15-mM MSA) - Pipet 1-mL of neat MSA for every liter of eluent to be made. Dilute MSA with filtered reagent water and store in appropriate sized carboys.

5.5.2 CSRS Cleaner Solution (1 M MSA) - Dilute 65-mL of MSA to 1-L with reagent water. Pour into a carboy. This is an alternate CSRS cleaner solution.

5.6 Nitric acid (HNO_3), 70%, Fisher Scientific

5.6.1 Cell Cleaning Solution (3 M HNO_3) - Dilute 22-mL of 70% HNO_3 (15 to 16 N) to 100-mL with reagent water in a volumetric flask.

5.7 Sulfuric Acid, concentrated, Trace Metals grade, Fisher Scientific

5.7.1 Cation Eluent (18 mN H_2SO_4) - Dilute 0.5-mL of concentrated sulfuric acid (36 N) to 1-L with reagent water. Pour into a carboy.

5.7.2 CSRS Cleaner Solution (1 N H_2SO_4) - Dilute 28-mL of concentrated sulfuric acid (36 N) to 1-L with reagent water. Pour into a carboy. This is the default CSRS cleaner solution.

5.8 Potassium chloride (KCl), Fisher Scientific

5.8.1 Cell Calibration Solution (0.001M KCl) - Prepare as follows:

5.7.1.1 Weigh out 0.7456-g of KCl into a 100-mL volumetric flask. Dilute with reagent water to the mark. This solution is 0.1M KCl.

5.7.1.2 Pipet 1.0-mL of the 0.1M KCl solution into a 100-mL volumetric flask and dilute with reagent water to the mark. This solution is 0.001 M KCl.

5.7.1.3 Use the 0.001M KCl solution for calibration of the cell, as necessary.

5.8 Sodium hydroxide (NaOH), Fisher Scientific

5.8.1 NaOH Solution (0.5M) - Weigh 2.0-g of NaOH into a 100-mL plastic volumetric flask, dissolve in and dilute with reagent water to 100-mL.

5.9 Hydrochloric acid (HCl), Fisher Scientific

5.9.1 Cation Column Cleaner solution (1N HCl) - Dilute 83-mL of the 37% concentrated HCl (11 to 12 N) to 1-L with reagent water. Pour into a carboy.

5.10 Cation Eluent (15-mM MSA)

5.10.1 18 mN sulfuric acid. This is the default eluent. See Section 5.7.1 for its preparation.

5.10.2 15 mM MSA. This is an alternate eluent. See Section 5.5.1 for its preparation.

5.11 Flush Solution - Reagent water is used as the flush solution.

5.12 Guanidine Nitrate (GN) Primary Standards - The primary standards are used for preparing initial (ICAL) and continuing calibration (CCVs) standard solutions, and spiking (LCS, and MS) solutions.

5.12.1 Stock Primary Solution (1,000-mg/L guanidinium) - Weight out 0.2032-g of GN from Aldrich into a 100-mL volumetric flask, dissolve in and dilute with reagent water to the mark. Use this stock solution for the preparation of the intermediate primary standard. This solution is good for six months when stored in a refrigerator.

5.12.2 Intermediate Primary Solution (100-mg/L guanidinium) - Pipet 10-mL of the Stock Solution (Section 5.12.1) into a 100-mL volumetric flask and dilute with reagent water to the mark. The eluent may be used instead of reagent water as a diluent for better ion chromatographic performance. This solution is good for one month when stored in a refrigerator.

5.12.3 Initial Calibration Standards (ICAL) - Pipet 1.0, 2.5, 5.0, 7.5, and 10-mL of the Intermediate Solution (Section 5.12.2) into 100-mL volumetric flasks and dilute with reagent water to the mark. The eluent may be used instead of reagent water as a diluent for better ion chromatographic performance. These ICAL solutions containing 1.0, 2.5, 5.0, 7.5, and 10.0-mg/L guanidinium ion are good for one week when stored in a refrigerator.

5.12.4 Continuing Calibration Standards (CCVs) - Pipet 5.0-mL of the Intermediate Solution (Section 5.12.2) into a 100-mL volumetric flask and dilute with reagent water to the mark. The eluent may be used

instead of reagent water as a diluent for better ion chromatographic performance. The CCV solution containing 5.0-mg/L guanidinium ion is good for one week when stored in a refrigerator. Use this solution to verify calibration daily before the analysis of environmental samples, at the end of analysis, and after every ten samples in between.

5.12.5 LCS and MS spiking solution (100-mg/L guanidinium) - This solution is identical to the Intermediate Solution (Section 5.12.2). This solution is good for one month when stored in a refrigerator.

5.13 Guanidine Nitrate (GN) Check Standards - The check standard is used exclusively for verifying the primary standard.

5.13.1 Stock Check Solution (1,000-mg/L guanidinium) - Weight out 0.2032-g of GN from ACROS into a 100-mL volumetric flask, dissolve in and dilute with reagent water to the mark. Use this stock solution for the preparation of the intermediate check standard. This solution is good for six months when stored in a refrigerator.

5.13.2 Intermediate Check Solution (100-mg/L guanidinium) - Pipet 10-mL of the Stock Check Solution (Section 5.13.1) into a 100-mL volumetric flask and dilute with reagent water to the mark. The eluent may be used instead of reagent water as a diluent for better ion chromatographic performance. This solution is good for one month when stored in a refrigerator.

5.13.3 Initial Calibration Verification Standard (ICV) - Pipet 5.0-mL of the Intermediate Check Solution (Section 5.13.2) into a 100-mL volumetric flask and dilute with reagent water to the mark. The eluent may be used instead of reagent water as a diluent for better ion chromatographic performance. The ICV solution containing 5.0-mg/L guanidinium ion is good for one week when stored in a refrigerator. Analyze this solution immediately after the analysis of initial calibration standards (Section 5.12.3) to check the accuracy of the primary standards.

5.14 Laboratory Control Samples (LCS) and LCS duplicates (LCSD) - Prepare LCS solutions in a manner similar to that described for CCV (Section 5.12.4).

5.15 Matrix Spike (MS) and MS duplicate (MSD) Samples - Prepare MS and MSD samples as described for LCS using the environmental sample instead of the eluent for dilution. The analyst may employ smaller volumes (e.g. spiking 50-uL of the Intermediate primary standard to 1.0-mL of sample) for the preparation of MS and MSD samples. Sample dilution should be taken into consideration while calculating percent recoveries.

5.16 Laboratory Blanks - Use the eluent (Section 5.10 or 5.5.1) as the sample for the analysis of method blanks (MB) and calibration blanks (CB, ICB, CCB, etc.).

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Oblong 4-Oz HDPE bottles (Level 6 precleaned) with foamed polyethylene lined polypropylene screw cap closures (QEC cat # 6212-0004) may be used. Provide 1 bottle for each water sample to be

collected. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.

6.2 Environmental samples should be analyzed within the holding times. The maximum holding time for GN is estimated at 6-months by Alliant TechSystems, Sunflower Army Ammunition Plant (SFAAP). Alliant TechSystems is an organization that has the most knowledge on the stability of GN and in-depth knowledge with this analysis. In the interim, the laboratory has adopted 28-days (commonly used for ion chromatography parameters) as the holding time for aqueous and soil samples.

6.3 One 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each solid sample. Quality control samples such as MS and MSD should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. These samples may be shared with samples collected for other analyses such as metals and organic parameters with the exception of VOCs.

7.0 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control may contain acids. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Soil samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters.

9.0 PROCEDURE

9.1 Calibration

9.1.1 Establish ion chromatographic operating parameters equivalent to those in the referenced method. Record the flow setting (approx. 1.25 ± 0.1 ml/min), pressure (approx. 1300 ± 200 psi) and conductivity (18 ± 2 μS) on the instrument maintenance log book. Discuss unusual conditions with the supervisor for appropriate resolution. Set up the data system as described in the data system manual. Use the same size loop for standards and samples. Use the autosampler for consistent results. Record chromatographic peak size in area units.

9.1.2 Prepare calibration standards at five concentration levels and a blank as described in Section 5. If the working range exceeds the linear range of the system, a sufficient number of standards must be analyzed to allow an accurate calibration curve to be established provided the system is operated on an applicable attenuator range. Unless the attenuator range settings are proven to be linear, each setting must be calibrated individually.

9.1.3 Make injections (volume determined by injection loop) of each initial calibration (ICAL) standard (Section 5.12.3). Tabulate peak area responses against the concentration. Calculate slope, y-intercept and correlation coefficient for linear regression from a plot of the peak areas against concentration. The correlation coefficient for the plot of response against concentration should be greater than 0.995 for the ICAL to be acceptable. During this procedure, retention times (RT) should also be recorded and RT windows calculated. Nonlinear response can result when the separator column capacity is exceeded (overloading).

9.1.4 The initial calibration curve (ICAL) must be verified with a second source standard (ICV) prepared as described in Section 5.13.3. Analyze this solution immediately after the analysis of initial calibration standards to check the accuracy of the primary standards. If the response varies from the expected value by more than ± 10%, the problem should be corrected (Check ICV and/or ICAL standard, instrument, etc.) and ICV analysis repeated, using a fresh ICV standard. If the results are still more than ± 10%, a new calibration curve (ICAL) should be prepared.

9.1.5 Continuing calibration verification (CCV) should be performed using the CCV standard solution (Section 5.12.4) to verify calibration daily before the analysis of environmental samples, at

the end of sample analysis, and after every ten samples in between. If the response for CCV varies from the expected value by more than ± 10%, the problem should be corrected (Check CCV standard, instrument, etc.) and CCV analysis repeated, using a fresh CCV standard. If the results are still more than ± 10%, associated samples since the last acceptable CCV should be reanalyzed.

9.2 Sample Preparation and Analysis

9.2.1 Aqueous Sample Preparation - Most aqueous samples (e.g. groundwater) do not require any preparation other than filtering them through 0.45-µm filter into an autosampler cup. For some aqueous samples a negative "water dip" is observed that may interfere with the analysis. The water dip, if present, may be removed by adding concentrated eluent (approximately 10-uL) to all samples (1-mL) and standards. Any dilutions that may be required in analyzing other water samples should be made with the eluent solution.

9.2.2 Soil Sample Preparation - For solid materials, the following extraction procedure should be used. Add an amount of reagent water equal to ten times the weight of solid material for soil sample extraction. Weigh out to soil (1-g) to three significant numbers, add 10-mL of reagent water, and mix the slurry for 10-minutes using a magnetic stirrer or a mechanical shaker. Allow the solid to settle down and filter the slurry using a 0.45 µ membrane type syringe filter. Analyze the aqueous extract as described for aqueous samples.

9.2.3 Ion Chromatographic Analysis of Samples

9.2.3.1 Start the flow of regenerant through the suppressor column. Set up the chromatography data system for maximum sensitivity and any additional ranges needed. Begin to pump the eluent through the columns. After a stable baseline is obtained, perform ICAL and ICV as described in Section 9.1.2 to 9.1.4 if necessary. If the instrument conditions have not changed since the last acceptable ICAL, proceed with CCV analysis (Section 9.1.5).

9.2.3.2 Inject the CCV standard solution. If the observed amount does not deviate by more than ±10% from the true value, proceed with CCB analysis. If the observed blank amount is less than one half of the method quantitation limit (MQL), proceed with MB analysis.

9.2.3.3 Inject a method blank (MB) sample. If the observed MB amount is less than one half of the method quantitation limit, proceed with LCS/LCSD sample analysis.

9.2.3.4 Prepare laboratory control samples (LCS/LCSD) as described in Section 5.14. Calculate the concentration from the calibration curve and compare it with the known value. The observed result should be within ±15% (85% <%R <115%) of the true value for aqueous samples. For soil samples, the observed result should be within ±30% (70% <%R <130%) of the true values. If the control limits are exceeded, stop the analysis until the problem is identified and corrected. Recalibration may be necessary. The RPD between LCS/LCSD results should be <20% for aqueous (<30% for soil) samples.

9.2.3.5 Prepare MS and MSD samples as described in Section 5.15. An unspiked sample and a spiked sample (MS and MSD) pair should be analyzed with each batch of samples. The observed MS/MSD recoveries (%R) should be within ±15% (85% <%R <115%) for aqueous samples. For soil samples, the observed recoveries should be within ±30% (70% <%R <130%). If the control limits are exceeded, flagging of the data may be necessary if corrective action procedures do not solve the problem. The RPD between LCS/LCSD results should be <20% for aqueous (<30% for soil) samples.

9.2.3.6 Analyze environmental samples, CCVs, and CCB (immediately after each CCV) as described earlier (Section 9.1.5). The MS and MSD samples should be treated as environmental samples while determining sample sequence.

9.2.3.7 The width of the retention time window used to make identifications should be based on measurements of actual retention time variations of standards over the course of a day. Use three times the standard deviation of the retention times to calculate the window size. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

9.2.3.8 If the response for the peak exceeds the working range of the system, dilute the sample with an appropriate amount of eluent and reanalyze. Perform 1:10 dilutions using class A volumetrics.

9.2.3.9 If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, spike the sample with an appropriate amount of standard and reanalyze.

9.3 Calculations

9.3.1 Compute sample concentration by comparing sample peak response with the standard curve. The HP Chemstation software can perform calibration and calculate sample results without the analyst performing any of the calculations shown below. The following procedure may be used to verify calculations performed by the instrument software.

9.3.2 Enter the calibration standard concentrations and peak areas from the data system into an Excel spreadsheet.

9.3.3 Calculate the following parameters: slope (m), intercept (c), and correlation coefficient (r). The slope and intercept define a relationship between the concentration and instrument response of the form:

$$y_i = m_i x_i + c \qquad (1)$$

where:
y_i = Instrument response (peak area)
m_i = response slope
x_i = concentration of standard i
c = intercept

Rearrangement of the above equation yields the concentration corresponding to an instrumental measurement:

$$x_j = (y_j - c)/m_j \qquad (2)$$

where:

x_j = calculated concentration for a sample
y_j = actual instrument response for a sample
m_j and c are calculated slope and intercept from calibration above.

9.3.4 Enter the sample peak areas into the spreadsheet, and calculate the sample concentration in milligrams per liter. Report guanidine nitrate as the guanidinium ion.

9.3.5 For solid samples, multiply the mg/L results obtained by the volume of sample extract (mL) and divide by the weight (g) of the solid sample used and the percent solids.

9.3.6 See AML SOP on training for the calculation of common environmental analytical parameters such as %R (for LCS and MS), RPDs, %D, etc.

10.0 QUALITY CONTROL

10.1 Quality control procedures are used to ensure that proper sample preparation techniques and instrument calibration procedures are performed. This involves the analysis of method blanks (MB), laboratory control samples (LCS), sample duplicates, and matrix spikes (MS).

10.2 The frequency of analysis of LCS analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples. LCS can be run in duplicate to obtain precision (RPD) information. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

10.3 The frequency of analysis of MS analysis is equivalent to a minimum of 2 per 20 samples or 2 per batch if less than 20 samples. MS is run in duplicate to obtain precision (RPD) information.

10.4 The frequency of analysis of MB analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples. The MS and MSD samples are counted as environmental samples. Laboratory QC samples such as MB, LCS, and LCSd are not counted as environmental samples as well as standards such as ICV, CCV, and CCB.

10.5 Include a continuing calibration verification standard (CCV) after each group of 10 samples in the analysis sequence for calibration check. The calculated values (or instrument response) for the calibration verification should be within 10 percent of the true values (or response from initial calibration). When this continuing calibration is out of this acceptance window, the laboratory should stop analyses and take corrective action.

10.6 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair.

10.6.1 The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on knowledge of the samples in the sample batch.

10.6.2 If samples are not expected to contain target analytes, the laboratory should use a matrix spike and matrix spike duplicate pair, spiked with the analyte at the mid-point of the initial calibration curve.

10.6.3 If samples are expected to contain target analytes, then the laboratory may use one matrix spike and a duplicate analysis of an unspiked field sample.

10.6.4 Since knowledge of the sample results may not always be available, AML should always analyze the MS in duplicate (MS and MSD).

10.6.5 Sample duplicate analyses need not be performed unless client requests laboratory duplicate analysis for specific projects. Duplicate analyses should be performed if the sample selected for MS contains large amounts of the target analyte(s).

10.7 Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

11.0DATA VERIFICATION AND VALIDATION - This section will provide guidance when evaluating and validating data generated by this SOP. It provides procedures to assess if data produced is scientifically valid, and legally defensible. Document review on the checklist (Appendix 7) and on the case narrative as appropriate.

11.1HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample preparation.

11.1.1 WATER - The referenced SOP from Alliant TechSystems describes the holding times as 6-months from the date of sample collection. A holding time of six months is common for elements (e.g. metals) and by extension to other cations such as guanidinium ions. Since the analyte is an organic cation, and it is not clear whether stability studies have been performed on the analyte, the laboratory will employ a holding time of 28-days, which is common for ion chromatographic analysis of stable anions in aqueous samples. Holding times are calculated in days from the date/time of sampling. Holding times are established by comparing the information in the chain of custody/sample log-in information with the date/time of the injection.

11.1.2 SOIL / SOLIDS– A 14-day holding is used for soil and waste samples for the preparation of sample extracts. Aqueous holding times (28-days) will apply to the sample extracts from the date of extract preparation. Holding times for soil sample extraction are calculated in days from the date of sampling.

11.1.3 ACTION – If holding times have been exceeded, contact the client. All positive results may require qualification (J or R depending on the client) by the laboratory or by the user depending on project. Include a statement in the case narrative indicating the outlier. The holding times may be subject to debate for this analyte.

11.2INITIAL CALIBRATION – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the IC is capable of producing data within a range that is linear. Document ICAL results in Form-6 equivalents (See Appendix 1 for details).

11.2.1 CHECK – Check to see that the target analyte has a correlation coefficient greater than 0.995.

11.2.2 ACTION – Prepare a fresh set of ICAL solutions and repeat the initial calibration procedure. If the situation cannot be corrected within the holding times of the samples, perform sample analysis. Positive results for compounds not meeting the criteria may need to be qualified as estimated (J) or rejected (R) either by the laboratory or the client depending on project). Describe outliners in the case narrative.

11.3 CALIBRATION VERIFICATION – Calibration verifications (ICV and CCVs) are used to check the integrity of the ICAL standards and to determine that the instrument is stable and operating under the linear range of the initial calibration. The ICV is prepared using a second source. The CCV is prepared from the same source as the primary standard. The calibration is verified after every 10 sample injections. Document ICV and CCV results in Form-7 equivalents (See Appendix 2 for details).

11.3.1 CHECK – Check to see that the observed ICV and/or CCV results are within ±10% of the true values.

11.3.2 ACTION – Correct the problem and rerun the calibration verification and all associated samples. Positive results for compounds not meeting the criteria may need to be qualified as estimated (J) or rejected (R) either by the laboratory or the client depending on project). Describe outliners in the case narrative.

11.4 METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Sample data qualification to alert clients about blank contamination is performed by the laboratory. Sample data qualification based on all available blank results (laboratory MB, equipment blank, and other field blanks) may be performed by the laboratory or the client depending on the project. The processing of blanks helps eliminate reporting of false positives in project reports.

11.4.1 Method blanks should be free of target analyte(s) and other interfering non-target analytes. To be acceptable, blank results should not exceed one half of the applicable method quantitation limit (MQL) for each analyte. The MQL (see Tables 1 and 2) for this analysis is 1.0-mg/L for aqueous samples and 10-mg/kg for soil/sediment samples.

11.4.2 Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples. If levels greater than one half the MQLs are present, inspect and correct the problem.

Reanalyze all associated samples. Contact client if other criteria are applicable. The only exception may be quick turn around projects.

11.4.3 ACTION – All positive results are reported with a "B" code, if contaminants are found in the method blank.

11.4.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, then no action is taken.

11.4.5 ACTION – Any compound detected in the sample which were also detected in associated method blank at levels greater than one half the PQLs should be re-extracted and/or re-analyzed unless other client criteria are applicable.

11.5 LABORATORY CONTROL STANDARD (LCS) – A LCS analysis is used to evaluate method competence in the absence of matrix effects. The LCS may provide a mechanism to check method efficiency for target analyte(s) without the sample matrix as a factor. Document LCS/LCSD results in Form-1 equivalents and recoveries in Form-3 equivalents (See Appendix 3 for details).

11.5.1 CHECK - Check the results for the LCS recoveries to see if the results are within the acceptable range (80% to 120% for water and 70% to 130% for soil) as shown in Tables 1 and 2. Flag outliers with an asterisk (*).

11.5.2 ACTION – Perform one manual calculation using raw data and extraction sheet information. If deviations are noticed, reanalysis of the LCS and associated samples will be necessary after correcting the problems.

11.5.3 The EPA methods do not require LCS to be run in duplicate. Therefore, QC limits for RPD for LCS and LCSD results (<20% for water and soil) is strictly advisory. However, if the RPD is not achieved on controlled samples, problems may be more severe with environmental sample duplicates and MS/MSD duplicates.

11.6 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike data are used to evaluate the precision and accuracy of the analytical method on project samples. Document MS/MSD results in Form-1 equivalents and recoveries in Form-3 equivalents (See Appendix 4 for details).

11.6.1 The QC limits for MS/MSD percent recoveries (70% to 130% for water and soil) and RPD (<30% for water and soil) are slightly wider than those for LCS and LCSD.

11.6.2 CHECK - Check the results for the MS/MSD recoveries with the applicable QC limits. Flag outliers with an asterisk (*).

11.6.3 Perform one manual calculation using raw data and extraction sheet information.

11.6.4 ACTION – No action is taken on MS/MSD recovery data alone to qualify associated samples for most clients. Data qualification

may be necessary for AFCEE projects. Since the presence of significant amounts of target and/or non-target anions and matrix interference could lead to lead to serious analytical problems, the supervisor should be consulted when significant outliers are noted. Document QC outliers for %recovery and RPD on the checklist and on client reports (*) and draw attention to the QC outliers on the case narrative. Professional judgment and/or procedures described in the applicable project plan may be used either by the laboratory or by the data user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries).

11.7 TARGET ANALYTE IDENTIFICATION AND REPORTING – Criteria for qualitative analysis is to minimize the number of erroneous positive or negative identifications of the analyte(s). Establish retention times and RT windows (See Appendix 5 for details). Report sample results in Form-1 equivalents (See Appendix 6 for details).

11.7.1 The retention time (RT) for positive identification of analyte(s) in samples should be within the RT windows established for each analyte.

11.7.2 ACTION – Professional judgement must be used when applying the RT window criteria since RT shifts are quite common due to significant difference between the sample and calibration media.

11.7.3 The MDL for guanidine nitrate is 0.136-mg/L in water and 0.14-mg/kg for soil/sediment samples (Tables 1 and 2). Report positive results that are greater than either the MDL and/or the method reporting limit (as appropriate) but lower than the method quantitation limit (MQL) as estimated with a "J" flag. Report results greater than the upper limit of calibration as estimated with an "E" flag. Such samples will require reanalysis with dilution. Both sets of results may need to be reported for some clients. See Tables 1 and 2 for a summary of the MDLs, RLs, MQLs, and control limits for QC samples. Adjust sample results and associated MDL, RL, and MQL, for percent moisture and non-standard sample weights.

12.0 REFERENCES

12.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9056, Determination of Inorganic Anions by Ion Chromatography, September 1994.

12.2 Determination of Guanidine Nitrate by Ion Chromatography, Alliant TechSystems, Sunflower Army Ammunition Plant, Operating Procedure Number HD-5-3-7210, Revision 6, 15-pages, April 2, 1996.

12.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.

12.4 Environmental Protection Agency. Test Method for the Determination of Inorganic Anions in Water by Ion Chromatography, Method 300.0. EPA-600/4-84-017, Revised document dated December 1989.

12.5 Annual Book of ASTM Standards, Volume 11.01 Water D4327, Standard Test Method for Anions in Water by Ion Chromatography, pp. 696-703. 1988.

12.6 Standard Methods for the Examination of Water and Wastewater, Method 4110B, "Determination of Anions by Ion Chromatography-Ion Chromatography with Chemical Suppression of Eluent Conductivity," 19th Edition, 1995.

12.7 Dionex, Operation and Maintenance Manual, Dionex Corp., Sunnyvale, CA 94086.

12.8 Method detection limit (MDL) as described in "Trace Analyses for Wastewater," J. Glaser, D. Foerst, G. McKee, S. Quave, W. Budde, Environmental Science and Technology, Vol. 15, Number 12, p. 1426, December 1981.

12.9 Gaskill, A.; Estes, E. D.; Hardison, D. L.; and Myers, L. E. Validation of Methods for Determining Chlorine in Used Oils and Oil Fuels. Prepared for U.S. Environmental Protection Agency Office of Solid Waste. EPA Contract No. 68-01-7075, WA 80. July 1988.

12.10 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

12.11 AML Standard Operating Procedures, Q-Water-RC.

12.12 AML Standard Operating Procedures, Q-WasteMgmt-RD.

12.13 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

12.14 AML Standard Operating Procedures, Q-ControlCharts-RC.

TABLE 1
Aqueous MDL, MQL and QC Limits for LCS, MS/MSD Analyses

Guanidine Nitrate Analysis by Ion Chromatography														
Analytical Management Laboratories														
Matrix: Water	Preparation Method			EPA-300.0/SW-9056				MDL Dates:			1/24/02	to	1/28/02	
	Analytical Method			EPA-300.0/SW-9056				IDP Dates:			1/24/02	to	1/28/02	
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**		**LCS Limits**		**MS Limits**		**RPD**
Analyte	Amount	Mg/L	mg/L	**Mg/L**	RD=B/A	flag	**mg/L**	RQ=C/A	flag	Lower	Upper	Lower	Upper	Limit
	mg/L	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%
Guanidinium	0.50	0.438	0.043	**0.136**	3.2		1.00	7.3		80	120	70	130	30
Mean Ratio (RD or RQ):					3.2			7.3						
NOTE: Guanidine Nitrate is reported as guanidinium ion in accordance with conventional practice.														
MDL= ST Dev * t factor (3.14 for 7 reps)														
MDLs and MQLs are subject to change due to ongoing method implementation changes.														
Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 5.														
Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.														
LCS and MS %R limits are laboratory limits with guidance from USACE EM200-1-3, Appendix I.														

TABLE 2
Soil/sediment MDL, MQL and QC Limits for LCS, MS/MSD Analyses

Guanidine Nitrate Analysis by Ion Chromatography														
Analytical Management Laboratories														
Matrix: Soil	Preparation Method			EPA-300.0/SW-9056				MDL Dates:						
	Analytical Method				EPA-300.0/SW-9056					IDP Dates:				
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**			**LCS Limits**		**MS Limits**	
Analyte	Amount	mg/kg	mg/kg	**mg/kg**	RD=B/A	Flag	**mg/kg**	RQ=C/A	flag	Lower	Upper	Lower	Upper	Limit
	mg/kg	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%
Guanidinium	5.0			**1.4**			10	7.3		70	130	70	130	30
Mean Ratio (RD or RQ):								7.3						
NOTE: Guanidine Nitrate is reported as guanidinium ion in accordance with conventional practice.														
MDL= ST Dev * t factor (3.14 for 7 reps). MDL was estimated for soil from aqueous data and the dilution factor (10).														
MDLs and MQLs are subject to change due to ongoing method implementation changes.														
Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 5.														
Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.														
LCS and MS %R limits are laboratory limits with guidance from USACE EM200-1-3, Appendix I.														

TABLE 3
List of Appendices

Appendix Number	Appendix Topic
1	Initial Calibration (ICAL) Summary (Form-6 Equivalents) Instrument Printouts
2	Initial Calibration Verification (ICV) Summary (Form-7 Equivalents) Continuing Calibration Verification (CCV) Summary (Form-7 Equivalents) Instrument Printouts
3	Laboratory Control Sample (LCS or LFB) Results (Form-1 Equivalents) LCS Recoveries Summary (Form-3 Equivalents)
4	Matrix Spike (MS) and MS Duplicate (MSD) Results (Form-1 Equivalents) MS/MSD Recoveries and RPD Summary (Form-3 Equivalents)
5	Retention Time Window Studies Summary
6	Sample Reports (Form-1 Equivalents)
7	Checklist for Data Review and Verification
8	Method Detection Limit (MDL) Studies Summary Miscellaneous Information

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices

STANDARD OPERATING PROCEDURES

No. I-Anions-IC-RC

DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPHY (EPA 300.0, 300.1 and SW-846 9056A)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPHY (EPA 300.0 and SW-846 9056A)

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) is based on SW-846 Method 9056A, EPA 300.0 and instrument manufacturer's recommended procedures.

1.1 This method addresses the sequential determination of the anions-chloride, fluoride, bromide, nitrate, nitrite, phosphate, and sulfate in aqueous samples (groundwater, drinking water, wastewater, etc) and the collection solutions from the bomb combustion of solid waste samples.

1.2 The method detection limit (MDL), the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero, varies for anions as a function of sample size and the conductivity scale used. Generally, minimum detectable concentrations are in the range of 0.05 mg/L for F^- and 0.1 mg/L for Br^-, Cl^-, $N0_3^-$, $N0_2^-$, PO_4^{3-}, and SO_4^{2-} with a 100-µL sample loop and a 10-µmho full-scale setting on the conductivity detector. Similar values may be achieved by using a higher scale setting and an electronic integrator. Idealized detection limits of an order of magnitude lower have been determined in reagent water by using a 1-µmho/cm full-scale setting (Table 4). The upper limit of the method is dependent on total anion concentration and may be determined experimentally. These limits may be extended by appropriate dilution.

2.0 SUMMARY OF METHOD

2.1 A small volume of the aqueous sample or the combustate collection solution, typically 1-mL, is injected into an ion chromatograph to flush and fill a constant volume sample loop. The sample is then injected into a stream of carbonate-bicarbonate eluent of the same strength as the collection solution or water sample.

2.2 The sample is pumped through three different ion exchange columns and into a conductivity detector. The first two columns, a precolumn or guard column and a separator column, are packed with low-capacity, strongly basic anion exchanger. Ions are separated into discrete bands based on their affinity for the exchange sites of the resin. The last column is a suppressor column that reduces the background conductivity of the eluent to a low or negligible level and converts the anions in the sample to their corresponding acids. The separated anions in their acid form are measured using an electrical-conductivity cell. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a calibration curve generated from known standards.

3.0 INTERFERENCES

3.1 Any species with retention times similar to that of the desired ion will interfere. Large quantities of ions eluting close to the ion of interest will also result in interference. Separation can be improved by adjusting the eluent concentration and/or flow rate. Sample dilution and/or the use of the method of standard additions can also be used. For example, high levels of organic acids may be present in industrial wastes, which may interfere with inorganic anion analysis. Two common species, formate and acetate, elute between fluoride and chloride.

3.2 Samples preserved with an acid such as sulfuric acid may cause severe interference for the analysis of other anions. If preservation is required to increase the holding times for anions such as nitrite and nitrate, the quantity of sulfuric acid should be tightly controlled to minimize the sulfate peak in the ion chromatogram.

3.3 Because bromide and nitrate elute very close together, they are potential interferences for each other. It is advisable not to have $Br^-/N0_3^-$ ratios higher than 1:10 or 10:1 if both anions are to be quantified. If nitrate is observed to be an interference with bromide, use of an alternate detector (e.g., electrochemical detector) is recommended.

3.4 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

3.5 Samples that contain particles larger than 0.45 µm and reagent solutions that contain particles larger than 0.20 µm require filtration to prevent damage to instrument columns and flow systems

3.6 If a packed bed suppressor column is used, it will be slowly consumed during analysis and, therefore, will need to be regenerated. Use of either an anion fiber suppressor or an anion micromembrane suppressor eliminates the time-consuming regeneration step through the use of a continuous flow of regenerant.

4.0 APPARATUS AND MATERIALS

4.1 Ion chromatograph, Dionex DX-120, capable of delivering 0.5 to 4.5 mL of eluent per minute and a maximum operating pressure of 4000 psi (28 MPa). The chromatograph is equipped with an injection valve, a 25- to 250-µL sample loop, and set up with the following components.

4.1.1 Precolumn, a guard column placed before the separator column to protect the separator column from being fouled by particulates or certain organic constituents (IonPac AG14 4-mm (10-32) Dionex P/N 46134).

4.1.2 Separator column, a column packed with low-capacity pellicular anion exchange resin that is styrene divinylbenzene-based has been

found to be suitable for resolving F^-, Cl^-, $N0_2^-$, $P0_4^{-3}$, Br^-, $N0_3^-$, and $S0_4^{-2}$ (AS14, 4-mm (10-32) 4 x 250 mm, Dionex P/N 46124).

4.1.3 Suppressor column, a column that is capable of converting the eluent and separated anions to their respective acid forms (ASRS Ultra 4-mm, Dionex P/N 53946).

4.1.4 Detector, a low-volume, follow-through, temperature-compensated, electrical conductivity cell (DS4 detector stabilizer, Dionex P/N 050218, approximately 1.25 µL volume) equipped with a meter capable of reading from 0 to 1,000 µseconds/cm on a linear scale.

4.1.5 Pump, capable of delivering a constant flow of approximately 0.5 to 4.5 ml/min throughout the test and tolerating a maximum pressure of 4000 psi (28 MPa).

4.1.6 Valco VICI digital valve sequence programmer for interfacing to the auto sampler.

4.1.7 Alcott 728 auto sampler

4.2 Hewlett Packard (HP) Chemstation chromatography data system equipped with HP 35900E A/D 2-channel interface.

4.3 Syringe, minimum capacity of 1 ml and equipped with a male pressure fitting.

4.4 Eluent and regenerant reservoirs, suitable containers for storing eluents and regenerant. Use 2-L plastic reservoirs (P/N 044129).

4.5 Analytical balance, capable of weighing to the nearest 0.0001 g.

4.6 Pipets, Class A volumetric flasks, beakers: assorted sizes.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Reagent water. See AML SOP on Reagent water (Q-Water-Rx) for additional details. Type I water should be used for the determination of the anions. Column life may be extended by passing reagent water through a 0.22-µm filter prior to use.

5.3 Eluent, 0.003M $NaHC0_3$/0.0024M Na_2C0_3. Dissolve 1.0080 g of sodium bicarbonate (0.003M $NaHC0_3$) and 1.0176 g of sodium carbonate (0.0024M Na_2C0_3) in reagent water and dilute to 4 L with reagent water.

5.4 Suppressor regenerant solution. Add 100 ml of 1N H_2S0_4 to 3 L of reagent water in a collapsible bag and dilute to 4 L with reagent water.

5.5 Stock solutions (1,000 mg/L). Stock solutions for each anion may be prepared as described below. Alternatively, a mixed stock solution containing anions of interest may be purchased as a certified solution from a suitable vendor (e.g. CPI).

5.5.1 Bromide stock solution (1.00 ml = 1.00 mg Br^-). Dry approximately 2 g of sodium bromide (NaBr) for 6 hours at 150°C, and cool in a desiccator. Dissolve 1.2877 g of the dried salt in reagent water, and dilute to 1 L with reagent water.

5.5.2 Chloride stock solution (1.00 ml = 1.00 mg Cl^-). Dry sodium chloride (NaCl) for 1 hour at 600°C, and cool in a desiccator. Dissolve 1.6484 g of the dry salt in reagent water, and dilute to 1 L with reagent water.

5.5.3 Fluoride stock solution (1.00 ml = 1.00 mg F^-). Dissolve 2.2100 g of sodium fluoride (NaF) in reagent water, and dilute to 1 L with reagent water. Store in chemical-resistant glass or polyethylene.

5.5.4 Nitrate stock solution (1.00 ml = 1.00 mg $N0_3^-$). Dry approximately 2 g of sodium nitrate ($NaN0_3$) at 105°C for 24 hours. Dissolve exactly 1.3707 g of the dried salt in reagent water, and dilute to 1 L with reagent water.

5.5.5 Nitrite stock solution (1.00 ml = 1.00 mg $N0_2^-$). Place approximately 2 g of sodium nitrate ($NaN0_2$) in a 125-ml beaker and dry to constant weight (about 24 hours) in a desiccator containing concentrated H_2S0_4. Dissolve 1.4998 g of the dried salt in reagent water, and dilute to 1 L with reagent water. Store in a sterilized glass bottle. Refrigerate and prepare monthly. Alternatively, order Nitrite (1000 ug/ml), Solution B of P/N 4400-010025, Multi-Element Ion Chromatography Standard 2 from CPI International.

NOTE: Nitrite is easily oxidized, especially in the presence of moisture, and only fresh reagents are to be used.

NOTE: Prepare sterile bottles for storing nitrite solutions by heating for 1 hour at 170°C in an air oven.

5.5.6 Phosphate stock solution (1.00 ml = 1.00 mg $P0_4^{3-}$). Dissolve 1.4330 g of potassium dihydrogen phosphate (KH_2P0_4) in reagent water, and dilute to 1 L with reagent water.

5.5.7 Sulfate stock solution (1.00 ml = 1.00 mg $S0_4^{2-}$). Dry sodium sulfate (Na_2S0_4) for 1 hour at 105°C and cool in a desiccator. Dissolve 1.4790 g of the dried salt in reagent water, and dilute to 1 L with reagent water.

5.5.8 Chlorate stock solution (1000 ug/ml), P/N 4400-010168 from CPI International.

5.5.9 Bromate stock solution (1000 ug/ml), P/N 4400-010169 from CPI International.

5.5.10 Chlorite stock solution (1000 ug/ml), P/N 4400-010170 from CPI International.

5.6 Mixed Calibration Stock Solutions: Order mixed calibration stock standard solutions containing nitrate-NO_3, phosphate-PO_4, fluoride, bromide, chloride, and sulfate-SO_4 (1000 ug/ml each, Solution A of P/N 4400-010025 multi-element ion chromatography standard 2) from CPI International. The anion concentrations are shown in Table 2A. If nitrates, nitrites and phosphates are reported in element units (N or P) the following conversion factors should be used:

1000 ppm Nitrate-NO_3 = 226 ppm Nitrate-N
1000 ppm Nitrite-NO_2 = 304 ppm Nitrite-N
1000 ppm Phosphate-PO_4 = 326 ppm Phosphate-P

5.7 Anion working solutions for initial calibration (ICAL). Prepare a blank and at least five different working solutions at the following anion concentrations (mg/L): 50, 20, 10, 5.0 and 2.5. Use the 10-mg/L solution for verification of the calibration (CALV). The ICAL anion solutions must be prepared in Class A volumetric flasks. See Tables 2B and 2C for details.

5.8 Anion working solution for calibration verification (CALV). Prepare a fresh 10-mg/L standard solution as described above for the verification of initial calibration. Prepare fresh everyday the analysis is performed. CALV solution may be prepared in smaller quantities (e.g. in 10-ml volumetric instead of 100-ml volumetric) using proportionately less amounts of the mixed stock solutions.

5.9 Laboratory Control Samples (LCS) and LCS duplicates (LCSD). Prepare LCS solutions in a manner similar to that described for CALV. Use standards with a lot number for LCS (e.g. 9BM125) which are different from those for calibration (e.g. 9EB084) if these are ordered from the same supplier (e.g. CPI).

5.10 Matrix Spike (MS) and MS duplicate (MSD) Samples. Prepare MS and MSD samples as described for LCS using the environmental sample instead of the eluent for dilution.

5.11 Stability of standards. Stock standards are stable for at least 1 month when stored at 4°C. Dilute working standards should be prepared weekly. The CALV and LCS should be prepared fresh daily.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Oblong 4-Oz HDPE bottles (Level 6 precleaned) with foamed polyethylene lined polypropylene screw cap closures (QEC cat # 6212-0004) may be used. Provide 1 bottle for each water sample to be

collected. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. If acid-preserved samples are collected for nitrate/nitrite analysis, an additional sample container without acid preservatives should be provided for collecting additional sample volumes for the analysis of other anions such as chloride, phosphate, and sulfate, if necessary.

6.2 Water samples should be analyzed within the holding times, which depends on the anion and the preservation method. See Table 3 for the holding times.

6.3 One 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each solid sample. Quality control samples such as MS and MSD should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. These samples may be shared with samples collected for other analyses such as metals and organic parameters with the exception of VOCs.

7.0 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control may contain acids. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Soil samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters.

9.0 PROCEDURE

9.1 Calibration

9.1.1 Establish ion chromatographic operating parameters equivalent to those that yield retention times shown in Table 1. Record the flow setting, pressure and conductivity on the instrument maintenance log book and discuss unusual conditions with the supervisor for appropriate resolution. The following operating conditions provide optimal results:
9.1.1.1 Flow Setting: 1.25 ± 0.2 mL/min.
9.1.1.2 Operating Pressure: 1800 ± 300 psi.
9.1.1.3 Conductivity: 18 ± 2 µS.

9.1.2 Set up the data system as described in the data system manual. For each anion of interest, prepare calibration standards at five concentration levels and a blank as described in Section 5. If the working range exceeds the linear range of the system, a sufficient number of standards must be analyzed to allow an accurate calibration curve to be established provided the system is operated on an applicable attenuator range. Unless the attenuator range settings are proven to be linear, each setting must be calibrated individually.

9.1.3 Make injections (volume determined by injection loop) of each calibration standard, starting with the highest calibration standard. Tabulate peak area responses against the concentration and prepare a calibration curve for each anion. For each anion, calculate slope, y-intercept and correlation coefficient for linear regression for a plot of the peak areas against concentration. Anions such as bromide, which are not commonly found in environmental samples, may be used as an internal standard. In such cases the response of the anion relative that of the internal standard should be plotted. Additionally, the concentration of the internal standard should remain constant in the initial calibration solutions. The correlation coefficient (r) for the plot of response against concentration should be greater than or equal to 0.995 for each anion for the ICAL to be acceptable. The coefficient of determination (COD) or coefficient of variation (r^2) should be ≥ 0.990. During this procedure, retention times (RT) should also be recorded and RT windows calculated.

9.1.4 Verify calibration initially (ICV) and throughout the analytical sequence as described below:

9.1.4.1 Initial calibration verification (ICV) is performed immediately after the analysis of ICAL solutions using a second source standard. If the response for any anion of interest varies from the expected values by more than ± 10%, corrective action will be necessary. Inspect the ICV solution first for accuracy of concentrations and integrity of the standards. Inspect the ICAL solutions, if no problems were identified with ICV. Replace ICAL and/or ICV, if necessary.

9.1.4.2 The working calibration curve must be verified on each working day (CCV), or whenever the anion eluent strength is changed, and for every batch of samples. If the response or retention time for any anion varies from the expected values by more than ± 10%, the test must be repeated, using fresh calibration standards. If the results are still more than ± 10%, a new calibration curve must be prepared for the applicable anion.

9.1.5 Nonlinear response can result when the separator column capacity is exceeded (overloading). Maximum column loading (all anions) should not exceed about 400 ppm.

9.2 Analyses

9.2.1 Sample preparation. Most aqueous samples such as groundwater samples do not require any preparation other than filtering them through 0.45-µm filter into an autosampler cup since they do not exhibit “water dip” (See below) that may interfere with fluoride and/or chloride analysis. When aqueous samples are injected, the water passes rapidly through the columns, and a negative "water dip" is observed that may interfere with the early-eluting fluoride and/or chloride ions. The water dip should not be observed in the combustate samples; the collecting solution is a concentrated eluent solution that will "match" the eluent strength when diluted to 100-mL with reagent water according to the bomb combustion procedure. Any dilutions that may be required in analyzing other water samples should be made with the eluent solution. The water dip, if present, may be removed by adding concentrated eluent to all samples and standards.

9.2.2 When a manual system is used, it is necessary to micropipet concentrated buffer into each sample. The recommended procedures follow: (1) Prepare a 100-mL stock of eluent 100 times normal concentration by dissolving 2.5202 g $NaHC0_3$ and 2.5438 g Na_2C0_3 in 100-mL reagent water. Protect the volumetric flask from air. (2) Pipet 5 ml of each sample into a clean polystyrene micro-beaker. Micropipet 50 µL of the concentrated buffer into the beaker and stir well. (3) Dilute the samples with eluent, if necessary, to concentrations within the linear range of the calibration.

9.2.3 For solid materials, the following extraction procedure should be used. Add an amount of reagent water equal to ten times the weight of solid material (weigh out to three significant numbers), e.g. 10-mL for 1-g sample. Mix the slurry for 10-minutes using a magnetic stirrer or a mechanical shaker. Allow the solid to settle down and filter the slurry using a 0.45 µ membrane type syringe filter. Analyze the aqueous extract as described for aqueous samples.

9.2.4 Sample Analysis

9.2.4.1 Start the flow of regenerant through the suppressor column. Set up the chromatography data system for maximum sensitivity and any additional ranges needed.

9.2.4.2 Begin to pump the eluent through the columns. After a stable baseline is obtained, inject a midrange standard (CALV) to verify initial calibration (ICAL). If the observed amount (or peak area) deviates by more than 10% from the true value (or peak area from that of the previous run), prepare fresh standards and reanalyze to verify initial calibration.

9.2.4.3 If a new initial calibration is required, begin to inject standards starting with the highest concentration standard and decreasing in concentration. Using the procedures described in Step 9.1, calculate the regression parameters (slope, y-intercept and correlation coefficient) for the initial standard curve. Compare these values with those obtained in the past. The correlation coefficient should be greater than 0.995 for each anion for the ICAL to be acceptable. If they exceed these limits, stop the analysis and look for the problem.

9.2.4.4 Inject a laboratory control sample (LCS). Calculate the concentration from the calibration curve and compare the known value. If the control limits are exceeded, stop the analysis until the problem is found. Recalibration is necessary.

9.2.4.5 A spiked sample (MS and/or MSD) should be analyzed with each batch of samples.

9.2.4.6 If internal standard procedure is used for initial calibration, fortify each sample with a fixed amount (e.g.10-ul per 1.0-ml sample) of an internal standard solution. The concentration of the internal standard used for spiking samples should be identical to that used in the calibration solutions. Load and inject a fixed amount of well-mixed sample. Flush injection loop thoroughly, using each new sample. Use the same size loop for standards and samples. Use the autosampler for consistent results. Record the resulting peak size in area units.

9.2.4.7 The width of the retention time window used to make identifications should be based on measurements of actual retention time variations of standards over the course of a day. Use three times the standard deviation of a retention time to calculate the window size for each anion. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

9.2.4.8 If the response for the peak exceeds the working range of the system, dilute the sample with an appropriate amount of eluent and reanalyze. Perform 1:10 dilutions using class A volumetrics.

9.2.4.9 If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, spike the sample with an appropriate amount of standard and reanalyze.

NOTE: Nitrate and sulfate exhibit the greatest amount of change, although all anions are affected to some degree. In some cases, this peak migration can produce poor resolution or misidentification.

9.3 Calculations

9.3.1 Prepare separate calibration curves for each anion of interest by plotting peak size in area units of standards against concentration values. Compute sample concentration by comparing sample peak response with the standard curve. The HP Chemstation software can perform calibration and calculate sample results without the analyst performing any of the calculations shown below. The following procedure may be used to verify calculations performed by the instrument software.

9.3.2 Enter the calibration standard concentrations and peak areas from the data system into an Excel spreadsheet.

9.3.3 Calculate the following parameters: slope (m), intercept (c), and correlation coefficient (r). The slope and intercept define a relationship between the concentration and instrument response of the form:

$$y_i = m_i x_i + c \qquad (1)$$

where:
y_i = Instrument response (peak area)
m_i = response slope
x_i = concentration of standard i
c = intercept

If internal standard calibration procedures are used, use relative responses as y_i or y_j in these equations. Rearrangement of the above equation yields the concentration corresponding to an instrumental measurement:

$$x_j = (y_j - c)/m_j \qquad (2)$$

where:

x_j = calculated concentration for a sample
y_j = actual instrument response for a sample
m_j and c are calculated slope and intercept from calibration above.

9.3.4 Enter the sample peak areas into the spreadsheet, and calculate the sample concentration in milligrams per liter. Report nitrite and nitrate as N. Report phosphate as P. Report chloride and fluoride as Cl and F respectively and sulfate as SO_4. Other anions such as bromate, chlorate, and chlorite should be reported as BrO_3, ClO_3, and ClO_2 respectively.

9.3.5 For solid samples, multiply the mg/L results obtained by the volume of sample extract (ml) and divide by the weight (g) of the solid sample used and the percent solids.

10.0 QUALITY CONTROL

10.1 Quality control procedures are used to ensure that proper sample preparation techniques and instrument calibration procedures are performed. This involves the analysis of method blanks (MB), laboratory control samples (LCS), sample duplicates, and matrix spikes (MS).

10.2 The frequency of analysis of LCS analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples. LCS can be

run in duplicate to obtain precision (RPD) information. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

10.3 The frequency of analysis of MS analysis is equivalent to a minimum of 2 per 20 samples or 2 per batch if less than 20 samples. MS is run in duplicate to obtain precision (RPD) information.

10.4 The frequency of analysis of MB analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples. Document MB association in Form-4 equivalents (See Appendix 10 for details). Include instrument run log and sample preparation log (for soil samples) in the data package.

10.5 Include a calibration standard after each group of 10 samples in the analysis sequence as a calibration check (CALV). The calculated values (or instrument response) for the calibration verification should be within 10 percent of the true values (or response from initial calibration). When this continuing calibration is out of this acceptance window, the laboratory should stop analyses and take corrective action.

10.6 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on knowledge of the samples in the sample batch. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair, spiked with the anions at the mid point of the initial calibration curve. If samples are expected to contain target analytes, then the laboratory may use one matrix spike and a duplicate analysis of an unspiked field sample. Since this knowledge may not always be available, AML will always analyze the MS in duplicate (MS and MSD). Sample duplicate analyses need not be performed unless client requests duplicate analysis for a specific project. Duplicate analyses should be performed if the sample selected for MS contains large amounts of the anion(s).

10.7 Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

11.0DATA VERIFICATION AND VALIDATION - This section will provide guidance and procedures to assess if data produced using this SOP are valid and legally defensible. Document review and comments on the checklist (See Appendix 6). Verify that sample preparation logs are complete and the instrument run logs are available for review (See Appendix 10). Anomalies and exceptions should be

documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on the checklists. Army (USACE) and Air Force (AFCEE) projects may have special requirements (See Appendix 5) that will require extra attention. A simple, universal procedure for evaluating and choosing appropriate corrective action doe not exist to deal with situations when one or more QC parameters are outside the acceptance window. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints.

11.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) or Air Force (AFCEE) projects may have additional requirements that are related to USACE laboratory validation (CX), USACE District requirements, government contract requirements, specific USACE project requirements or USACE Architect/Engineer (A/E) requirements. USACE-CX requirements (Summary of Method Quality Objectives for Method 9056) are included in Appendix 5.

11.1.1 The USACE requirements shown in Appendix 5 and elsewhere in this SOP are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may or may not be attainable for routine sample analysis. Since USACE MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

11.1.2 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality objectives and/or other QC requirements. Exceptions and data qualifications necessary should be documented on the checklists and eventually on case narratives.

11.1.3 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist, perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst

review is complete. For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete. For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director will review the checklists (initial/date as necessary), prepare case narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

11.1.4 The QC limit for method blank is one half of the applicable MQL (default) or one-half of the applicable reporting limits (project specific).

11.2 HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

11.2.1 WATER - The holding times are shown in Table 3. Holding times are calculated in days and/or hours from the date/time of sampling. Holding times are established by comparing the information in the chain of custody/sample log-in information with the date/time of the injection.

11.2.2 SOIL / SOLIDS– A 14 day holding is used for soil and waste samples for the preparation of sample extracts. Aqueous holding times will apply to the sample extracts. Holding times for sample extraction are calculated in days and/or hours from the date/time of sampling.

11.2.3 ACTION – If holding times have been exceeded, contact the client. All positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or invalid (I).

11.3 INITIAL CALIBRATION (ICAL) and INITIAL CALIBRATION VERIFICATION (ICV) – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the IC is capable of producing acceptable data within a calibration range of interest for the parameter. A second source ICV is used to verify the accuracy of calibration standards and QC samples prepared from the primary stock standards. Document ICAL results in Form-6 equivalents, linear regression graphs, etc. (See Appendix 1 for more details). Document ICV (See Appendix 5 for details) results in Form-6 equivalents as described for CCVs.

11.3.1 Check – Inspect the calibration data for the target anions and verify that the correlation coefficients (r) are less than or equal to 0.995 or that the coefficient of determinations (COD or r^2) are less than or equal to 0.99.

11.3.2 ACTION – Prepare a fresh set of ICAL solutions and repeat the initial calibration procedure. Adjust instrument parameters as necessary to improve sensitivity if that is a problem. If the situation cannot be corrected within the holding times of the samples, perform sample analysis using an alternate method, if possible.

11.3.3 Check – Inspect the ICV data. The %D for ICV should be within ±10% for the anions of interest.

11.3.4 ACTION – Check ICV solution first and the ICAL solutions for accuracy and integrity. Replace one or both as necessary and repeat ICAL and ICV.

11.3.5 ACTION - Positive results for compounds not meeting the criteria may need to be qualified as estimated (J) by the user (client). Document the anomaly in the case narrative.

11.4 CONTINUING CALIBRATION – The continuing calibration is used to check that the instrument is still operating under the linear range of the initial calibration and has remained stable. The calibration is verified after every 10 sample injections. Document CALV results in Form-7 equivalents (See Appendix 2 for more details).

11.4.1 Anions – Check to see that the observed values for each anion is within 10% or less of the true values. If internal standards are used, check to see that the internal standard response has not changed by more than ± 50% from the response obtained during initial calibration (NOTE: This is AML QC requirement).

11.4.2 ACTION – Correct the problem and rerun the calibration verification and all associated samples. Positive results for compounds not meeting the criteria may be qualified as estimated (J) by the user (client). Report non-detects without qualifiers and mention these outliners in the case narrative.

11.5 METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Sample data qualification to alert clients about blank contamination is performed by the laboratory. Sample data qualification based on all available blank results (laboratory MB, equipment blank, and other field blanks) is performed by the user (client). The processing of blanks helps eliminate reporting of false positives in project reports.

11.5.1 Method blanks should be free of all target anaytes and other interfering non-target analytes. To be acceptable, blank results should not exceed the QC limit, which is one half of the applicable method quantitation limit (default) or one half of the reporting limit (project specific) for each anion. See Tables 4 and 5 for a list of MQLs.

11.5.2 Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples. If levels greater than one half the MQLs are present, inspect and correct the problem. Reanalyze all associated samples. Contact client if other criteria are applicable. The only exception may be quick turn around projects.

11.5.3 ACTION – All positive results are reported with a "B" code, if contaminants are found in the method blank.

11.5.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, then no action is taken.

11.5.5 ACTION – Any compound detected in the sample which were also detected in associated method blank at levels greater than one half the MQLs should be re-extracted and/or re-analyzed unless other client criteria are applicable.

11.6 LABORATORY CONTROL STANDARD (LCS) – A LCS analysis is used to evaluate method competence in the absence of matrix effects. The LCS should be from a second source standard. The LCS may provide a mechanism to check method efficiency for target compounds without the sample matrix as a factor. Document LCS/LCSD results in Form-1 equivalents as described for sample results and recoveries in Form-3 equivalents (See Appendix 4 for details).

11.6.1 LCS recovery limits are shown in Tables 4 and 5.

11.6.2 Check the results for the LCS recoveries to see if the results are within the acceptable range. Flag outliers with an asterisk (*).

11.6.3 ACTION – No action is taken on LCS recovery data alone to qualify all associated samples. A general rule is to have all the anions that are contaminants of concern for specific projects to be within control limits. The supervisor should be consulted if this is not achievable for specific projects due to the short holding times for sample analysis. If significant deviations are noticed, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements. Check with the project manager about specific project requirements. No action may need to be taken on LCS and/or LCSD recovery data alone either to reanalyze samples and/or to qualify associated samples for most projects. Professional judgment may be used by the user in evaluating sample data qualification options in conjunction with other QC data for the project.

11.7 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike data are used to evaluate the precision and accuracy of the analytical method on project samples. Document MS/MSD results in Form-1 equivalents as described for sample results and recoveries in Form-3 equivalents (See Appendix 3 for details).

11.7.1 MS/MSD percent recovery limits and the limits for relative percent difference (RPD) between the MS and MSD recoveries are shown in Tables 4 and 5.

11.7.2 Check the results for the MS/MSD recoveries with the applicable QC limits. Flag outliers with an asterisk (*).

11.7.3 Perform one manual calculation using raw data and extraction sheet information.

11.7.4 ACTION – No action is taken on MS/MSD recoveries data alone to qualify all associated samples. A general rule is to have all the anions within the control limits. Since the presence of significant amounts of target and/or non-target anions and matrix interference could lead to lead to serious analytical problems, the supervisor should be consulted when significant outliers are noted. Document QC outliers for %recovery and RPD on the checklist and on client reports (*) and draw attention to the QC outliers on the case narrative. No action may be taken on MS/MSD recovery data alone either to reanalyze samples and/or to qualify associated samples either by the analyst or by the end user. Professional judgment may be used by the user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

11.8 TARGET ANION IDENTIFICATION – Criteria for qualitative analysis is to minimize the number of erroneous positive or negative identifications of the anions. Document MDL studies (See Appendix 8 for details) RT windows in logbooks (See Appendix 7 for details) and other applicable files.

11.8.1 The retention time (RT) for positive identification of anions in samples should be within the RT windows established for each anion.

11.8.2 ACTION – Professional judgement must be used when applying the RT window criteria since RT shifts are quite common due to significant difference between the sample and calibration media.

11.8.3 Report positive results that are greater than either the MDL and/or the reporting limit (as appropriate) but lower than the MQL as estimated with a "J" flag. Report results greater than the upper limit of calibration with as estimated with an "E" flag. Such samples will require reanalysis with dilution. Both sets of results

may need to be reported for some clients. See Tables 4 and 5 for a summary of the MDLs, RLs and MQLs. Adjust sample results and associated MDL, RL, and MQL, for percent moisture and non-standard sample weights.

12 REFERENCES

12.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9056, Determination of Inorganic Anions by Ion Chromatography, September 1994.

12.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9056A, Determination of Inorganic Anions by Ion Chromatography, Update IV-B.

12.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.

12.4 Environmental Protection Agency. Test Method for the Determination of Inorganic Anions in Water by Ion Chromatography, Method 300.0. EPA-600/4-84-017, Revised document dated December 1989.

12.5 Environmental Protection Agency. Determination of Inorganic Anions in Drinking Water by Ion Chromatography, Method 300.1, 1997, Errata, April 1999.

12.6 Annual Book of ASTM Standards, Volume 11.01 Water D4327, Standard Test Method for Anions in Water by Ion Chromatography, pp. 696-703. 1988.

12.7 Standard Methods for the Examination of Water and Wastewater, Method 4110B, "Determination of Anions by Ion Chromatography-Ion Chromatography with Chemical Suppression of Eluent Conductivity," 19th Edition, 1995.

12.8 Dionex, Operation and Maintenance Manual, Dionex Corp., Sunnyvale, CA 94086.

12.9 Method detection limit (MDL) as described in "Trace Analyses for Wastewater," J. Glaser, D. Foerst, G. McKee, S. Quave, W. Budde, Environmental Science and Technology, Vol. 15, Number 12, p. 1426, December 1981.

12.10 Gaskill, A.; Estes, E. D.; Hardison, D. L.; and Myers, L. E. Validation of Methods for Determining Chlorine in Used Oils and Oil Fuels. Prepared for U.S. Environmental Protection Agency Office of Solid Waste. EPA Contract No. 68-01-7075, WA 80. July 1988.

12.11 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

12.12 AML Standard Operating Procedures, Q-Water-RC.

12.13 AML Standard Operating Procedures, Q-WasteMgmt-RD.

12.14 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

12.15 AML Standard Operating Procedures, Q-ControlCharts-RC.

TABLE 1

CHROMATOGRAPHIC CONDITIONS

ANION	RETENTION TIME (RT) Range (Minutes)
Fluoride	2.87 to 3.16
Chloride	3.85 to 4.40
Nitrite	4.50 to 5.20
Bromide	5.42 to 6.42
Nitrate	6.18 to 7.40
Sulfate	8.20 to 9.80
Phosphate, ortho	10.45 to 13.00

Standard conditions:
Columns/sample loop and detector - As specified in Section 4 and 9.1.1.

[a]The retention time given for each anion is based on equipment and analytical conditions described in this SOP. Use of other analytical columns or different elutant concentrations will affect retention times accordingly.

TABLE 2-A
STOCK CALIBRATION SOLUTION

Anion	Concentration CPI-4400-010025-A (mg/L)	Concentration CPI-4400-010025-B (mg/L)
Fluoride (F^-)	1000	
Chloride (Cl^-)	1000	
Nitrite ($N0_2^-$) or (Nitrite-N)		1000 (304)
Bromide (Br^-)	1000	
Phosphate ($P0_4^{3-}$) or (Phosphate-P)	1000 (326)	
Nitrate ($N0_3^-$) or (Nitrate-N)	1000 (226)	
Sulfate ($S0_4^{2-}$)	1000	

TABLE 2-B
PREPARATION OF STANDARD SOLUTIONS FOR INITIAL CALIBRATION

ICAL Solution Number	ICAL Anion ($N0_3$, $N0_2$, $P0_4$, Cl, etc.) Concentration (mg/L)	Volume of CPI-4400-010025-A (ml)	Volume of CPI-4400-010025-B (ml)	Volume Of Eluent (ml)	Total Volume (ml)
ICAL-5	50	5.0	5.0	90.0	100
ICAL-4	20	2.0	2.0	96.0	100
ICAL-3	10	1.0	1.0	98.0	100
ICAL-2	5	0.5	0.5	99.0	100
ICAL-1	2.5	0.25	0.25	99.5	100
ICAL-0	0.0	0.0	0.0	100.0	100

TABLE 2-C
CONCENTRATIONS (mg/L) OF ICAL SOLUTIONS

Anion	ICAL-5	ICAL-4	ICAL-3	ICAL-2	ICAL-1
Fluoride (F^-)	50	20	10	5	2.5
Chloride (Cl^-)	50	20	10	5	2.5
Nitrite ($N0_2^-$) or (Nitrite-N)	50 15.2	20 6.08	10 3.04	5 1.52	2.5 0.76
Bromide (Br^-)	50	20	10	5	2.5
Phosphate ($P0_4^{3-}$) or (Phosphate-P)	50 16.3	20 6.52	10 3.26	5 1.63	2.5 0.815
Nitrate ($N0_3^-$) or (Nitrate-N)	50 11.3	20 4.52	10 2.26	5 1.13	2.5 0.565
Sulfate ($S0_4^{2-}$)	50	20	10	5	2.5

NOTE: ICAL-3 is used to verify initial calibration.

TABLE 3
HOLDING TIMES

Anion	Aqueous Samples Preserved at 4°C	Aqueous Samples Preserved With acid	Soil Samples Preserved at 4°C Extraction
Nitrate	48-hours	28-days	14-days
Nitrite	48-hours	28-days	14-days
Chloride	28-days	28-days	14-days
Sulfate	28-days	28-days	14-days
Phosphate	48-hours	28-days	14-days
Fluoride	28-days	28-days	14-days
Bromide	28-days	28-days	14-days

TABLE 4
Aqueous MDLs, MQLs and QC Limits for LCS/LCSD and MS/MSD

Anions Analysis by Ion Chromatography														
Analytical Management Laboratories														
Matrix: Water	Preparation Method			EPA300/SW9056		MDL Date:			10/29/01					
	Analytical Method			EPA300/SW9056										
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	Ratio (RQ)		**LCS Limits**		**MS Limits**		**RPD**
Anion	Amount	Mg/L	mg/L	**mg/L**	RD=B/A	flag	**mg/L**	RQ=C/A	flag	Lower	Upper	Lower	Upper	Limit
	mg/L	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%
Fluoride	0.1	0.14	0.01	**0.042**	3.2		0.20	4.7		85	115	80	120	20
Chloride	0.1	0.29	0.04	**0.12**	2.4		1.00	8.1		85	115	80	120	20
Nitrite	0.03	0.06	0.02	**0.061**	1.0		0.30	4.9		85	115	80	120	20
Bromide	0.10	0.16	0.04	**0.130**	1.2		1.00	7.7		85	115	80	120	20
Nitrate	0.023	0.04	0.01	**0.022**	1.8		0.23	10.3		85	115	80	120	20
Phosphate	0.326	0.32	0.03	**0.106**	3.1		0.33	3.1		85	115	80	120	20
Sulfate	0.1	0.20	0.03	**0.09**	2.3		1.00	11.2		85	115	80	120	20
Mean Ratio (RD or RQ):					2.1			7.2						
MDL= ST Dev * t factor (3.14 for 7 reps)														
MDLs and MQLs are subject to change due to ongoing method implementation changes.														
Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 5.														
Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.														
LCS and MS %R limits are laboratory limits with guidance from USACE EM200-1-3, Appendix I.														

TABLE 5
Soil/sediment MDLs, MQLs and QC Limits for LCS/LCSD and MS/MSD

Anions Analysis by Ion Chromatography														
Analytical Management Laboratories														
Matrix: Water	Preparation Method			EPA300/SW9056		MDL Date:			10/29/01					
	Analytical Method			EPA300/SW9056										
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	Ratio (RQ)		**LCS Limits**		**MS Limits**		**RPD**
Anion	Amount	mg/kg	mg/kg	**mg/kg**	RD=B/A	flag	**mg/kg**	RQ=C/A	flag	Lower	Upper	Lower	Upper	Limit
	mg/kg	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%
Fluoride	1.0	1.4	0.10	**0.42**	3.2		0.20	4.7		85	115	70	130	30
Chloride	1.0	2.9	0.40	**1.2**	2.4		1.00	8.1		85	115	70	130	30
Nitrite	0.30	0.60	0.20	**0.61**	1.0		0.30	4.9		85	115	70	130	30
Bromide	1.0	1.6	0.40	**1.3**	1.2		1.00	7.7		85	115	70	130	30
Nitrate	0.23	0.40	0.10	**0.22**	1.8		0.23	10.3		85	115	70	130	30
Phosphate	3.3	3.2	0.30	**1.1**	3.1		0.33	3.1		85	115	70	130	30
Sulfate	1.0	2.0	0.30	**0.90**	2.3		1.00	11.2		85	115	70	130	30
Mean Ratio (RD or RQ):					2.1			7.2						
MDL= ST Dev * t factor (3.14 for 7 reps) - Soil MDLs are estimated from aqueous data and the dilution factor (10).														
MDLs and MQLs are subject to change due to ongoing method implementation changes.														
Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 10.														
Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.														
LCS and MS %R limits are laboratory limits with guidance from USACE EM200-1-3, Appendix I.														

TABLE 6
List of Appendices

Appendix Number	Appendix Topic
1	Initial Calibration (ICAL) Summary (Form-6 equivalents) Instrument Printouts
2	Calibration Verification (ICV and CCV) Summary (Form-7 equivalents) Instrument Printouts and Standard Chromatograms
3	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
4	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
5	Client Method Quality Objectives for Method 9056 (USACE, AFCEE, etc.)
6	Checklist for Review and Data Verification
7	Retention Time Window Studies Summary
8	Method Detection Limit (MDL) Study Summary
9	Sample Reports (Form-1 equivalents)
10	QC Association Forms (Form-4 equivalents) Sample Preparation Logsheets Instrument Run Log

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. I-3010APrep-RD

ACID DIGESTION OF AQUEOUS SAMPLES FOR TOTAL METALS ANALYSIS BY FLAA or ICP-AES

October 21, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

ACID DIGESTION OF AQUEOUS SAMPLE FOR TOTAL METALS ANALYSIS BY FLAA or ICP-AES

1.0 Scope and Application

1.1 This standard operating procedure (SOP) is based on EPA SW-846 Method 3010A with modifications based on Method 3005, EPA 200.7, EPA ILM 04.0, ILM 05.0 and ILM 05.2. This SOP describes acid digestion procedures used to prepare aqueous samples, TC and TCLP leachates, and aqueous waste containing suspended solids for analysis by flame atomic absorption spectroscopy (FLAA) or inductively coupled argon plasma atomic emission spectroscopy (ICP-AES).

1.2 Prior to using this SOP, employees performing this preparation are encouraged to become familiar with underlying EPA methods to be sure they have a complete understanding of the method guidelines.

1.3 The digestates from this procedure are not suitable for analysis using graphite furnace atomic absorption spectroscopy (GFAA) or inductively coupled argon plasma mass spectrometry (ICP-MS).

1.4 Samples prepared by this method may be analyzed by FLAA or ICP-AES for all the metals indicated below.

Aluminum	Lead
Antimony	Magnesium
Arsenic	Manganese
Barium	Molybdenum
Beryllium	Nickel
Cadmium	Potassium
Calcium	Selenium
Chromium	Silver
Cobalt	Sodium
Copper	Thallium
Iron	Vanadium
	Zinc

2.0 Summary of Procedure

2.1 A representative volume (50-mL) of sample is digested by refluxing in nitric acid and hydrochloric acid and made up to a final volume (50-mL) equal to the initial sample volume.

3.0 Interferences

3.1 Aqueous samples containing significant amounts of sludge may present its own challenge during sample digestion and analysis.

3.2 Procedures contained in this SOP have been optimized to accommodate a diverse group

of metals - to minimize loss from volatilization during sample preparation and to maximize matrix spike recoveries.

3.3 The hot plate temperature should be monitored to prevent boiling and evaporation to dryness. Samples should be covered to avoid the introduction of contamination.

4.0 Apparatus and Materials

4.1 Hot Block Digester (Environmental Express).

4.2 50-mL disposable digester cups (Environmental Express) with 10-ml, 25-ml and 50-ml calibration marks. Verify calibration as described in AML SOP on volumetric measurements.

4.3 Digester caps, disposable (Environmental Express)

4.4 Filters, disposable (Environmental Express)

4.5 Ribbed watch glasses for use with digester cups, disposable (Environmental Express)

5.0 Reagents and Standards

5.1 ASTM Type I water: See AML SOP on reagent water.

5.2 Concentrated nitric acid, reagent grade (HNO_3): Acid should be analyzed to determine levels of impurities. If method blank is <MDL, the acid can be used.

5.3 Concentrated Hydrochloric acid, reagent grade. Hydrochloric acid should be analyzed to determine level of impurities. If method blank is <MDL, the acid can be used. Hydrochloric acid is used when the method is modified to include the analysis of mercury by ICP-MS. See sample preparation logs (Figure 1) for quantities used.

5.4 LCS Spiking Solution: See SOP on the determinative procedures (e.g. 61010 SOP) for details on preparing the spiking solution. Spike 1-ml of the ICPWS Water Spiking Solution and 1-ml of the ICPAgS Water and Soil Spike Solution to each clean matrix.

5.4.1 Preparation of the LCS sample. Pour 50-mL of reagent water to the mark in the sampling cup. Pipette in 1-ml of the ICPWS and 1-ml of the ICPAgS solution to the reagent water. Put the cap on and invert the sample cup a few times for adequate mixing. Prepare LCS duplicate (LCSD) using an identical procedure. Process these samples as any other environmental sample using procedures described in Section 9.

5.5 Matrix Spike (MS) Spiking Solutions: Use the solutions and quantities listed in Section 5.4 for spiking actual environmental samples.

5.5.1 Preparation of the MS sample. Pour 50-mL of the sample designated for matrix spike analysis to the mark in the sampling cup. Pipette in 1-ml of the ICPWS and 1-ml of the ICPAgS solution to the sample. Put the cap on and invert the sample cup a few times for adequate mixing. Prepare MS duplicate (MSD) using an identical procedure. Process these samples as any other environmental sample using procedures described in Section 9.

5.6 Method Blank. Pour 50-mL of reagent water to the mark in the sampling cup. Process this sample as any other environmental sample using procedures described in Section 9.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 For definitions of terms, sample collection and Preservation: See Table 1.

6.1.1 Total Metals: The concentration of metals determined in a sample following digestion.
6.1.2 Suspended Metals: The concentration of metals determined in the portion of a sample that has been filtered through 2.0 um filters and followed by digestion.
6.1.3 Dissolved Metals: The concentration of metals determined in a sample after the sample is filtered through a 0.45 um filter.
6.1.4 Total recoverable metals: The concentration of metals in an unfiltered sample following treatment with hot dilute mineral acid.

6.2 One liter plastic bottles with screw caps may be used for collecting aqueous samples in accordance with EPA SW-846 methods 6010B and 7000 series methods. Order pre-cleaned bottles containing the nitric acid preservative directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each water sample to be collected. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. See project specific field sampling plan (FSP) for sample collection and preservation instructions. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.
6.3 Field filtration of samples should be encouraged for dissolved metals analysis. If filtration cannot be performed in the field, provide 1 bottle not containing the acid preservative for each water sample to be collected. See Section 6.2 for additional containers for QA/QC samples.
6.4 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Analytical data generated outside of the specified holding times must be considered to be minimum values only. The holding times for the extraction and/or analysis of aqueous and soil samples (including TCLP leachate preparations) are 6-months from the date of sample collection.

7.0 HEALTH AND SAFETY

7.1 Standards used for calibration or spiking may contain a number of toxic heavy metals at high concentrations. The toxicity of these complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.
7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.
7.3 Reagents used such as acids are highly corrosive. Gloves and safety glasses should be worn to prevent contact with skin and eyes.
7.4 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.
7.5 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.
8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.
8.3 Soil samples that have undergone extraction should be free of contaminants. They can be disposed off into sanitary dumpsters. Dispose of sample extracts containing high levels of acid in accordance with applicable AML SOPs.

9.0 PROCEDURE

Record measurements, observations, etc. on the sample preparation sheets contained in bound logbooks (Appendix 1). Record the lot numbers of all the reagents and standards used in sample preparation. Two separate digestion procedures are described. The first procedure (Section 9.5) labeled as "SW 3010" may be used if low MS/MSD recoveries for antimony that are common for this digestion method are acceptable for the project. The second procedure (Section 9.6) labeled as "EPA CLP" may be used if antimony recoveries are required to be within stated QC limits. EPA uses this method to report total metals from all aqueous samples, which include groundwater, wastewater, and surface water.
NOTE: Since antimony in aqueous samples are analyzed using other instrumental procedures (GFAA and ICP-MS), the SW 3010 digestion procedure is the preferred method.
NOTE: Clean Water Act does not recognize ICP-MS methods and State Regulators may reject data from ICP-AES methods not meeting LCS recovery requirements.
NOTE: Sections 9.1 to 9.4 described below are common to both procedures.

9.1 Retrieve samples to be analyzed from the cooler. Shake the sample thoroughly to homogenize any fine sediment within the sample container. If pH of the sample is unavailable (check with sample log-in data), determine pH using either a probe or paper and verify the sample has been properly preserved. If the sample is not preserved at a pH <2, check with the project manager for specific instructions. Add the appropriate amount of acid prior to sample digestion, if necessary and record the quantity and lot number of the acid used to adjust the pH. This ensures the acids used in the digestion do not lose their strength.
9.2 Place clean, labeled Environmental Express hot block sample digestion cups alongside the aqueous samples requiring digestion.
9.3 If the pH is <2, carefully pour 50-mL of the sample into the Environmental Express sample digestion cup to the mark. Follow the analytical protocol specified in the analytical method to be used make up the necessary quality control samples. A method blank (MB), lab control sample (LCS), lab control sample duplicate (LCSD), matrix spike (MS), and matrix spike duplicate (MSD) should be prepared along with every batch of samples or at a rate of at least 5%. See individual determinative analytical methods and project specific quality assurance project plans (QAPP) for specific quality control requirements.
9.4 At this time, all spike aliquots should be added to the appropriate QC samples. Spike each LCS, LCSD, MS and MSD sample with 1-mL each of the ICPWS and ICPAgS spiking solutions. The samples are now ready for digestion according to Section 9.5 or 9.6.

9.5 SW 3010 Procedure

9.5.1 Add 1.5-mL of concentrated HNO_3 to the cups (Section 9.4). Cover the digestion cups with a ribbed covers and place them on the hot block maintained at 90° C to 95° C and cautiously evaporate to a low volume (2- to 3-mL), making certain that the sample does not boil and that no portion of the bottom of the sample cups are allowed to go dry. NOTE: If a sample is allowed to go to dryness, low recoveries will result. Should this occur, discard the sample and repeat sample digestion using fresh aliquots as outlined in Sections 9.1 to 9.4.

9.5.2 Cool the cups and add another 1.5-mL portion of concentrated HNO_3. Cover the cups with a non-ribbed covers and return to the hot block. Increase the temperature of the hot block, if necessary, so that a gentle reflux action occurs.

9.5.3 Continue heating, adding additional acid as necessary, until the digestion is complete, which is generally indicated when the digestate is light in color or does not change in appearance with continued refluxing. Again, uncover the cup or use a ribbed cover, and evaporate to a low volume (1- to 2- mL), not allowing any portion of the bottom of the cup to go dry.

9.5.4 Cool the beaker. Add 5-mL of 1:1 HCl. Cover the cup, and reflux for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

9.5.5 Wash down the cup walls and the covers with Type-1 water, adjust the volume to 50-mL with Type-1 water. Cap and shake the sample digestate and allow them to stand for approximately 30-mintes so that the particulates can settle down. When necessary, insert an Environmental Express filter cartridge into the sample tubes to remove silicates and other insoluble materials that could clog the instrument nebulizer. NOTE: Most samples do not need this filtration step. For some USACE samples, centrifuging may be necessary instead of the separation procedure described above. If it is required, place tubes in centrifuge for 15 minutes to spin down sediment and heavier particulates.

9.5.6 The sample is now ready for analysis.

9.6 EPA CLP Procedure

9.6.1 Add 1-mL of 1:1 HNO_3 and 5.0-mL of 1:1 HCl to the cups (Section 9.4).

9.6.2 Place the digestion cups on the hot block maintained at 90° C to 95° C and cautiously evaporate for 2 hours or until the samples are reduced to a low volume (10- to 25-mL), making certain that the sample does not boil and that no portion of the bottom of the cup is allowed to go dry.

9.6.3 Remove samples from the hotblock and cool to room temperature.

9.6.4 After samples have cooled, rinse the cup walls with Type I water and bring the final volume to the 50-mL graduation mark with Type I water. Cap and shake the sample digestate and allow them to stand for approximately 30-mintes so that the particulates can settle down.

9.6.5 NOTE: Filtration may be necessary for samples containing excessive

particulates or silicates. If the digestates are highly turbid, insert an Environmental Express filter cartridge into the sample tubes to separate the particulates from the digestate. NOTE: For some USACE samples, centrifuging may be necessary instead of the separation procedure described above. If it is required, place tubes in centrifuge for 15 minutes to spin down sediment and heavier particulates.

9.6.6 Sample digestates are now ready for instrumental analysis.

10.0 REFERENCES

10.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 6010B, Inductively Coupled Plasma Atomic Emission Spectrometry, December 1996.

10.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 6010C, Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 3, November 2000.

10.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 3.2, Sample Preparation Methods, December 1996.

10.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 3.3, Methods for Determination of Inorganic Analytes, December 1996.

10.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3010A, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy, July 1992.

10.6 Methods for the Chemical Analysis of Water and Wastes, US EPA, EPA-600/4-79-020, Inductively Coupled Plasma, Method 200.7.

10.7 Methods for the Determination of Metals in Environmental Samples, US EPA, EPA-600/4-91-01020, June 1991, Method 200.7, Determination of Metals and Trace Elements in Water and Wastes by Inductively Couples Plasma-Atomic Emission Spectrometry, p-31.

10.8 USEPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, ILM04.0, April 1994.

10.9 EPA Contract Laboratory Program SOW ILM05.0, Exhibit D.

10.10 EPA Contract Laboratory Program SOW ILM05.2, Page D-16/ICP-AES, December 2001.

10.11 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

10.12 AML Standard Operating Procedures, Q-Water-RC.

10.13 AML Standard Operating Procedures, Q-Subsampling-RC.

10.14 AML Standard Operating Procedures, Q-WasteMgmt-RD.

10.15 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

10.16 AML Standard Operating Procedures, Q-Controlcharts-RC.

Table 1

SAMPLE HOLDING TIMES, DIGESTED VOLUMES AND COLLECTION VOLUMES FOR METALS DETERMINATIONS IN AQUEOUS AND SOLID SAMPLES

Measurement	Digestion Amount	Sample Collection Volume or Weight	Holding Time Preservation (4°C)
Total	50-mL	600-mL	6 months, HNO3 to pH <2
Dissolved	50-mL	600-mL	6 months, filter on site; HNO3 to pH <2
Suspended	50-mL	600-mL	6 months, filter on site
Solid (Total)	1.25-g	200-gm	6 months

Figure 1

Sample Preparation Log

Analytical Management Laboratories, Inc. - Olathe, KS

ICP WATER - METALS Digestion Log

MDIG Batch # :				LCS & MS Spike Solution Names :	ICPWS and ICPAgS
META Batch # :				LCS & MS Spike Solution Reference #'s :	
Digestion Method :				LCS & MS Volumes :	
Digestion Date :				Spike Witness :	
Analyst Initials :		Reviewed By :		HNO3 Lot # :	
Time Start :				HCl Lot # :	
Time Stop :				Temperature :	

CLIENT SAMPLE NUMBER	LABORATORY SAMPLE NUMBER	Initial Sample Volume (mL)	Digestate Final Volume (mL)	Dilution Factor	REMARKS
NA	MB	(50)	(50)	1	
NA	LCS	(50)	(50)	1	
NA	LCSD	(50)	(50)	1	

STANDARD OPERATING PROCEDURES

No. I-3020APrep-RD

ACID DIGESTION OF AQUEOUS SAMPLES FOR TOTAL RECOVERABLE METALS ANALYSIS BY GFAA or ICP-MS

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

ACID DIGESTION OF AQUEOUS SAMPLE FOR TOTAL RECOVERABLE METALS ANALYSIS BY GFAA or ICP-MS

1.0 Scope and Application

1.1 This standard operating procedure (SOP) is based on EPA SW-846 Method 3020A, 3005, 7060, 7740, EPA 200.8, 200.9, EPA ILM 04.0, ILM 05.0, ILM05.1, and ILM 05.2. This SOP describes acid digestion procedures used to prepare aqueous samples for analysis by the graphite furnace atomic absorption spectroscopy (GFAA) or inductively coupled argon plasma spectroscopy mass spectrometry (ICP-MS).

1.2 Prior to using this SOP, employees performing this preparation are encouraged to become familiar with underlying EPA methods to be sure they have a complete understanding of the method guidelines.

1.3 This procedure is used to determine total recoverable metals that could become environmentally available. This procedure is not a total digestion technique and samples prepared using minor modifications of this method (described in this SOP) may be analyzed for mercury by ICP-MS.

1.4 Sample digestates prepared by this SOP may be analyzed for TAL metals by GFAA (except mercury) or ICP-MS (no exceptions). See determinative methods for details.

2.0 Summary of Procedure

2.1 A representative volume (50-ml) of sample is digested by refluxing in nitric acid and hydrogen peroxide. Dilute nitric acid is employed as the final dilution acid for the GFAA and/or ICP-MS analysis.

3.0 Interferences

3.1 Aqueous samples containing significant amounts of sludge may present its own challenge during sample digestion and analysis.

3.2 Procedures contained in this SOP have been optimized to accommodate a diverse group of metals such as antimony, arsenic, selenium, etc. to minimize loss from volatilization during sample preparation and to maximize matrix spike recoveries.

3.3 The hot plate temperature should be monitored to prevent boiling and evaporation to dryness. Samples should be covered to avoid the introduction of contamination.

4.0 Apparatus and Materials

4.1 Hot Block Digester (Environmental Express).

4.2 50-mL disposable digester cups (Environmental Express) with 10-ml, 25-ml and 50-ml calibration marks. Verify calibration as described in AML SOP on volumetric measurements.

4.3 Digester caps, disposable (Environmental Express)

4.4 Filters, disposable (Environmental Express)
4.5 Ribbed watch glasses for use with digester cups, disposable (Environmental Express)

5.0 Reagents and Standards

5.1 ASTM Type I water: See AML SOP on reagent water.
5.2 Concentrated nitric acid, reagent grade (HNO_3): Acid should be analyzed to determine levels of impurities. If method blank is <MDL, the acid can be used.
5.3 Concentrated Hydrochloric acid, reagent grade. Hydrochloric acid should be analyzed to determine level of impurities. If method blank is <MDL, the acid can be used. Hydrochloric acid is used when the method is modified to include the analysis of mercury by ICP-MS. See sample preparation logs (Figure 1) for quantities used.
5.4 Hydrogen Peroxide, 30% reagent grade (H_2O_2): Reagent should be analyzed to determine levels of impurities. If method blank is <MDL, the reagent can be used.
5.5 LCS Spiking Solution: See SOP on the determinative procedures (GFAA or ICPMS SOP) for details on preparing the spiking solution. Spike 1-ml of the GFAASS (or ICPMS as appropriate) Water and Soil Spike Solution to each clean matrix.
5.5.1 Preparation of the LCS sample. Pour 50-mL of reagent water to the mark in the sampling cup. Pipette in 1-ml of the GFASS to the reagent water. Put the cap on and invert the sample cup a few times for adequate mixing. Prepare LCS duplicate (LCSD) using an identical procedure. Process these samples as any other environmental sample using procedures described in Section 9.

5.6 Matrix Spike (MS) Spiking Solutions: Use the solutions and quantities listed in Section 5.5 for spiking actual environmental samples.
5.6.1 Preparation of the MS sample. Pour 50-mL of the sample designated for matrix spike analysis to the mark in the sampling cup. Pipette in 1-ml of the GFASS solution to the sample. Put the cap on and invert the sample cup a few times for adequate mixing. Prepare MS duplicate (MSD) using an identical procedure. Process these samples as any other environmental sample using procedures described in Section 9.
5.7 Method Blank. Pour 50-mL of reagent water to the mark in the sampling cup. Process this sample as any other environmental sample using procedures described in Section 9.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 For definitions of terms, sample collection and Preservation: See Table 1.

6.1.1 Total Metals: The concentration of metals determined in a sample following digestion.
6.1.2 Suspended Metals: The concentration of metals determined in the portion of a sample that has been filtered through 2.0 um filters and followed by digestion.
6.1.3 Dissolved Metals: The concentration of metals determined in a sample after the sample is filtered through a 0.45 um filter.
6.1.4 Total recoverable metals: The concentration of metals in an unfiltered sample following treatment with hot dilute mineral acid.

6.2 One liter plastic bottles with screw caps may be used for collecting aqueous samples in accordance with EPA SW-846 methods 6010B and 7000 series methods. Order pre-cleaned bottles containing the nitric acid preservative directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each water sample to be collected. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. See project specific field sampling plan (FSP) for sample collection and preservation instructions. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.

6.3 Field filtration of samples should be encouraged for dissolved metals analysis. If filtration cannot be performed in the field, provide 1 bottle not containing the acid preservative for each water sample to be collected. See Section 6.2 for additional containers for QA/QC samples.

6.4 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Analytical data generated outside of the specified holding times must be considered to be minimum values only. The holding times for the extraction and/or analysis of aqueous and soil samples (including TCLP leachate preparations) are 6-months from the date of sample collection.

7.0 HEALTH AND SAFETY

7.1 Standards used for calibration or spiking may contain a number of toxic heavy metals at high concentrations. The toxicity of these complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Reagents used such as acids are highly corrosive. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.4 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.5 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Soil samples that have undergone extraction should be free of contaminants. They can be disposed off into sanitary dumpsters. Dispose of sample extracts containing high levels of acid in accordance with applicable AML SOPs.

9.0 PROCEDURE

Record measurements, observations, etc. on the sample preparation sheets contained in bound logbooks (Figure 1). Record the lot numbers of all the reagents and standards used in sample preparation. Two

separate EPA digestion procedures are described. Sections 9.1 to 9.4 are common to both procedures The first procedure (Section 9.5) labeled as "SW-846 3020A" may be used if 3020A is the required sample digestion procedure and/or sample digestates are analyzed by graphite furnace atomic absorption (SW-846 7000) methods. The second procedure (Section 9.6) labeled as "EPA 200.8/CLP" may be used for all other applications that involves ICP/MS analysis. EPA uses the 200.8/CLP method to report total metals from all aqueous samples, which include groundwater, wastewater, and surface water from ICP/MS analysis.

9.1 Retrieve samples to be analyzed from the cooler. Shake the sample thoroughly to homogenize any fine sediment within the sample container. If pH of the sample is unavailable (check with sample log-in data), determine pH using either a probe or paper and verify the sample has been properly preserved. If the sample is not preserved at a pH <2, check with the project manager for specific instructions. Add the appropriate amount of acid prior to sample digestion, if necessary. This ensures the acids used in the digestion do not lose their strength.

9.2 Place clean, labeled Environmental Express hot block sample digestion cups alongside the aqueous samples requiring digestion.

9.3 If the pH is <2, carefully pour 50-mL of the sample into the Environmental Express sample digestion cup to the mark. Follow the analytical protocol specified in the analytical method to be used make up the necessary quality control samples. A method blank (MB), lab control sample (LCS), lab control sample duplicate (LCSD), matrix spike (MS), and matrix spike duplicate (MSD) should be prepared along with every batch of samples or at a rate of at least 5%. See individual determinative analytical methods and project specific quality assurance project plans (QAPP) for specific quality control requirements.

9.4 At this time, all spike aliquots should be added to the appropriate QC samples. Spike each LCS, LCSD, MS and MSD sample with 1-ml of GFAASS Water and Soil Spiking solution.

9.5 SW-846 3020A Procedure

9.5.1 Add 1.5 mL of concentrated HNO_3. Cover with a ribbed watch glass. Place the cups on the hot block maintained at 90°C to 95° C and cautiously evaporate to a low volume (2 to 3 mL), making certain that the sample does not boil and that the cups do not dry up. **NOTE:** If a sample is allowed to go to dryness, low recoveries will result. Should this occur, discard the sample and repeat sample digestion using fresh aliquots as outlined in Sections 9.1 to 9.4.

9.5.2 Cool the sample cups and add another 1.5 mL portion of concentrated HNO_3. Cover with a non-ribbed watch glass. Return to the hot block and gently reflux the samples.

9.5.3 Continue heating, adding additional acid as necessary, until the digestion is complete, which is generally indicated when the digestate is light in color or does not change in appearance with continued refluxing. Again, uncover the cup or use a ribbed cover, and evaporate to a low volume (1 to 2 mL), not allowing any portion of the bottom of the cup to go dry. See note in Section 9.5.1.

9.5.4 Remove the sample cups and add 10 mL of Type-1 water, mix, and continue heating the samples on the hot block for 10 to 15 minutes to allow additional solubiliztion of any residue to occur.

9.5.5 Remove the sample cups from the hot block and wash down the cup walls and the covers with Type-1 water, adjust the volume to 50-mL with Type-1 water. Cap

and shake the sample digestate and allow them to stand for approximately 30-minutes so that the particulates can settle down.

9.5.6 When necessary, insert an Environmental Express filter cartridge into the sample tubes to remove silicates and other insoluble materials that could clog the ICP/MS instrument nebulizer. **NOTE:** Most samples do not need this filtration step. For some USACE samples, centrifuging may be necessary instead of the separation procedure described above. If it is required, place tubes in centrifuge for 15 minutes to spin down sediment and heavier particulates.

9.5.7 The sample digestates are now ready for analysis by the chosen determinative method (GFAA or ICP/MS).

9.6 EPA 200.8/CLP Procedure

9.6.1 Add 1.0 mL of 1:1 HNO_3 and 0.5 mL of 1:1 HCl to the sample cups (see Section 9.4).

9.6.2 Cover the digestion cups with a ribbed covers and place the digestion cups on the hot block maintained at 85° C and cautiously evaporate for 2 hours or until the samples are reduced to a low volume (10 mL), making certain that the samples do not boil and that no portion of the bottom of the cup is allowed to go dry.

9.6.3 Remove the ribbed watch glass covers and cover with non-ribbed watch glass. Reflux the samples gently for 30 minutes making certain that the samples do not boil and that no portion of the bottom of the cup is allowed to go dry.

9.6.4 Remove samples from the hotblock and cool to room temperature.

9.6.5 After samples have cooled, rinse the cup walls with Type I water and bring the final volume to the 50-mL graduation mark with Type I water. Cap and shake the sample digestate and allow them to stand for approximately 30-mintes so that the particulates can settle down.

9.6.6 **NOTE:** Filtration may be necessary for samples containing excessive particulates or silicates. If the digestates are highly turbid, insert an Environmental Express filter cartridge into the sample tubes to separate the particulates from the digestate.

9.6.7 **NOTE:** For some USACE samples, centrifuging may be necessary instead of the separation procedure described above. If it is required, place tubes in centrifuge for 15 minutes to spin down sediment and heavier particulates.

9.6.8 Sample digestates are now ready for instrumental analysis.

10.0 REFERENCES

10.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 6020A, Inductively Coupled Plasma-Mass Spectrometry, January 1998.

10.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 7000A, Atomic Absorption Methods, July 1992.

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10.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual

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10.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 3.2, Sample Preparation Methods, December 1996.

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10.8 Methods for the Chemical Analysis of Water and Wastes, US EPA, EPA-600/4-79-020, AA Furnace Methods for Antimony (204.2), Arsenic (206.2), Cadmium (213.2), Chromium (218.2), Lead (239.2), Selenium (270.2), and Thallium (279.2).

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10.11 EPA Contract Laboratory Program SOW ILM05.0, Exhibit D.

10.12 EPA Contract Laboratory Program SOW ILM05.1, Exhibit D.

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10.14 USEPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, ILM04.0, April 1994.

10.15 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

10.16 AML Standard Operating Procedures, Q-Water-RC.

10.17 AML Standard Operating Procedures, Q-Subsampling-RC.

10.18 AML Standard Operating Procedures, Q-WasteMgmt-RD.

10.19 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

10.20 AML Standard Operating Procedures, Q-Controlcharts-RC.

Table 1

SAMPLE HOLDING TIMES, DIGESTED VOLUMES AND COLLECTION VOLUMES FOR METALS DETERMINATIONS IN AQUEOUS AND SOLID SAMPLES

Measurement	Digestion Amount	Sample Collection Volume or Weight	Holding Time* Preservation (4°C)
Total	50-mL	600-mL	6 months, HNO3 to pH <2
Dissolved	50-mL	600-mL	6 months, filter on site; HNO3 to pH <2
Suspended	50-mL	600-mL	6 months, filter on site
Solid (Total)	1.25-g	200-gm	6 months

NOTE: * The holding time for mercury (ICP-MS analyte) is 28-days.

Figure 1

Sample Preparation Logs

Analytical Management Laboratories, Inc. - Olathe, KS
ICP-MS and GFAA WATER - METALS Digestion Log

MDIG Batch # : _____

LCS & MS Spike Solution Names : ICPMSS1 & ICPMSS2

META Batch # : _____

LCS & MS Spike Solution Reference #(s) :

Digestion Method : AML-3020-R

LCS & MS Volumes : 0.1 mL of each

Digestion Date : _____

Spike Witness :

Analyst Initials : _____ Reviewed By : _____

HNO_3 Lot # : (0.5 mL)

Time Start : _____

H_2O_2 Lot # : (1.0 mL)

Time Stop : _____ Temp. _____

HCl Lot # : (0.2 mL)

CLIENT SAMPLE NUMBER	LABORATORY SAMPLE NUMBER	Initial Sample Volume (mL)	Digestate Final Volume (mL)	Dilution Factor	REMARKS
NA	MB	(50)	(50)	1	
NA	LCS	(50)	(50)	1	
NA	LCSD	(50)	(50)	1	

STANDARD OPERATING PROCEDURES

No. I-3050BPrep-RD

ACID DIGESTION OF SOIL, SEDIMENTS AND SLUDGES FOR TOTAL RECOVERABLE METALS ANALYSIS

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

ACID DIGESTION OF SEDIMENTS, SLUDGES, AND SOILS FOR ELEMENTAL ANALYSIS

1.0 Scope and Application

1.1 This standard operating procedure (SOP) is based on EPA SW-846 Method 3050B, EPA 200.7, 200.8, 200.9, EPA ILM 04.0, ILM 05.0, and other commonly accepted sample preparation procedures. This SOP describes three acid digestion procedures used to prepare sediments, sludges, and soil samples for analysis by the following analytical methods: (a) flame atomic absorption spectroscopy (FLAA) or inductively coupled plasma atomic emission spectroscopy (ICP-AES), and (b) graphite furnace atomic absorption spectroscopy (GFAA) or inductively coupled argon plasma spectroscopy mass spectrometry (ICP-MS).

1.2 Prior to using this SOP, employees performing this preparation are encouraged to become familiar with underlying EPA methods to be sure they have a complete understanding of the method guidelines.

1.3 This procedure is used to determine total recoverable metals that could become environmentally available. This procedure is not a total digestion technique since elements bound in silicate structures are not normally dissolved by this procedure. Samples prepared by this method may be analyzed by ICP for all the listed metals or by FLAA or GFAA as indicated below.

FLAA / ICP-AES or ICP-MS		GFAA
Aluminum	Magnesium	Antimony
Antimony	Manganese	Arsenic
Arsenic	Molybdenum	Cadmium
Barium	Nickel	Chromium
Beryllium	Potassium	Lead
Cadmium	Selenium	Selenium
Calcium	Silver	Thallium
Chromium	Sodium	
Cobalt	Thallium	
Copper	Vanadium	
Iron	Zinc	
Lead		

2.0 Summary of Procedures

2.1 3050-Hard Digestion Procedure: A representative 1- to 2-g (wet weight) sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with either nitric acid or hydrochloric acid. Dilute (1:1) hydrochloric acid is used as the final reflux acid for (1) the GFAA analysis of Sb and (2) the ICP analysis of Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Ag, Na, Tl, V, and

Zn. Dilute nitric acid is employed as the final dilution acid for the furnace AA analysis of As, Cd, Cr, Pb, Se, and Tl. A separate sample should be dried for a total solids determination.

2.2 3050-Soft Digestion Procedure: A 1:1 mixture of concentrated nitric acid and hydrochloric acids are used for this procedure. Hydrogen peroxide, which has a tendency to reduce antimony recoveries, is omitted for this variation.

2.3 3050-PBM Digestion Procedure: A 1:3 mixture of concentrated nitric acid and hydrochloric acids are used for this procedure. Silver is more readily solubilized and it tends to stay in solution at the higher hydrochloric acid levels employed in this procedure.

3.0 Interferences

3.1 Sludge samples may be diverse matrix types, each of which may present its own challenge during sample digestion and analysis.

3.2 Volatilization of antimony and other metals can occur with hard digestion procedures that may be required for some projects.

3.3 Precipitation of silver can occur with the hard and soft digestion procedures described in this SOP. Performance based methods (PBM) may be required for projects that require adherence to QC limits for antimony and silver.

3.4 The hot plate temperature should be monitored to prevent boiling and evaporation to dryness. Samples should be covered to avoid the introduction of contamination.

4.0 Apparatus and Materials

4.1 Hot Block Digester (Environmental Express).

4.2 50-mL disposable digester cups (Environmental Express) with 10-ml, 25-ml and 50-ml calibration marks. Verify calibration as described in AML SOP on volumetric measurements.

4.3 Digester caps, disposable (Environmental Express)

4.4 Filters, disposable (Environmental Express)

4.5 Ribbed watch glasses for use with digester cups, disposable (Environmental Express)

4.6 Analytical balance (Ohaus) capable of 0.01-g or higher level of accuracy.

5.0 Reagents and Standards

5.1 <u>ASTM Type I water</u>: See AML SOP on reagent water.

5.2 <u>Concentrated nitric acid</u>, reagent grade (HNO_3): Acid should be analyzed to determine levels of impurities. If method blank is <MDL, the acid can be used.

5.3 <u>Concentrated Hydrochloric acid</u>, reagent grade and 1:1 HCl, prepared from Type I water and conc. hydrochloric acid. Hydrochloric acid should be analyzed to determine level of impurities. If method blank is <MDL, the acid can be used.

5.4 <u>Aqua Regia:</u> Mix three volumes of concentrated hydrochloric acid (e.g. 90-mL) with one volume of concentrated nitric acid (30-mL) in a 200-mL beaker to prepare 120-mL of Aqua Regia. Prepare fresh immediately before sample digestion. Prepare 5-mL for each sample that requires digestion including QC samples (MB, LCS, LCSD, MS,

MSD, etc.).

5.5 Hydrogen Peroxide, 30% reagent grade (H_2O_2): Reagent should be analyzed to determine levels of impurities. If method blank is <MDL, the reagent can be used.

5.6 LCS Spiking Solution (FLAA/ICP-AES): See SOP on the determinative procedures (e.g. 6010 SOP) for details on preparing the spiking solution. Spike 1-ml of the ICPSS Soil Spike Solution and 1-ml of the ICPAgS Soil and Water Spike Solution to each clean matrix.

5.6.1 Preparation of the LCS sample. Pour 50-mL of reagent water to the mark in the sampling cup. Pipette in 1-ml of the ICPSS and 1-ml of the ICPAgS solution to the reagent water. Put the cap on and invert the sample cup a few times for adequate mixing. Prepare LCS duplicate (LCSD) using an identical procedure. Process these samples as any other environmental sample using procedures described in Section 9.

5.7 LCS Spiking Solution (GFAA/ICP-MS): See SOP on the determinative procedures (e.g. 7000 SOP) for details on preparing the spiking solution. Spike 1-ml of the GFAASS Water and Soil Spike Solution to each clean matrix.

5.7.1 Preparation of the LCS sample. Pour 50-mL of reagent water to the mark in the sampling cup. Pipette in 1-ml of the GFASS to the reagent water. Put the cap on and invert the sample cup a few times for adequate mixing. Prepare LCS duplicate (LCSD) using an identical procedure. Process these samples as any other environmental sample using procedures described in Section 9.

5.8 Matrix Spike (MS) Spiking Solutions: Use the solutions and quantities listed in Sections 5.5 and 5.6 for spiking actual environmental samples.

5.8.1 Prepare MS and MSD samples using procedures described for the LCS and LCSD substituting reagent water with the sample designated for MS/MSD analysis.

5.9 Method Blank. Pour 50-mL of reagent water to the mark in the sampling cup. Process this sample as any other environmental sample using procedures described in Section 9.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Sample collection and Preservation: See Table 1. One 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples in accordance with EPA SW-846 Method 6010B or 7000 series methods. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.).

6.2 Provide 1 bottle for each solid sample. Quality control samples such as MS and MSD should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. These samples may be shared with samples collected for other analyses such as chlorinated pesticides (8081A), PCBs (8082), other pesticides (8140, etc.), semivolatile organics (8270C) and diesel range organics (DRO, 8015-mod.) or TPH.

6.3 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Analytical data generated outside of the specified holding times must be considered to be minimum values only.

The holding times for the extraction and/or analysis of aqueous and soil samples (including TCLP leachate preparations) are 6-months from the date of sample collection.

7.0 HEALTH AND SAFETY

7.1 Standards used for calibration or spiking may contain a number of toxic heavy metals at high concentrations. The toxicity of these complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.
7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.
7.3 Reagents used such as acids and acid mixtures are highly corrosive. Gloves and safety glasses should be worn to prevent contact with skin and eyes.
7.4 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors. Preparation of aqua regia should be performed in a fume hood to minimize exposure to nitrous fumes.
7.5 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.
8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.
8.3 Soil samples that have undergone extraction should be free of contaminants. They can be disposed off into sanitary dumpsters. Dispose of sample extracts containing high levels of acid in accordance with applicable AML SOPs.

9.0 PROCEDURE

Record measurements, observations, etc. on the sample preparation sheets contained in bound logbooks (Appendix 1). Record the lot numbers of all the reagents and standards used in sample preparation. Two separate digestion procedures are described. The first procedure labeled as "Hard Digestion" may be used if low MS/MSD recoveries for antimony are acceptable for the sake of getting slightly higher sample results for other target elements. The second procedure labeled as "Soft Digestion" may be used if antimony recoveries are required to be within stated QC limits. The third procedure labeled as "PBM Digestion" may be used if silver recoveries are required to be within QC limits. The Metals Team Leader is responsible for directing appropriate digestion procedures that should be used for specific projects. Steps 9.1 to 9.4 are common for all these procedures.

9.1 Place clean, Environmental Express hot block sample digestion cups along with samples next to the weighing station. Mix the sample thoroughly with a spatula to achieve homogeneity and sieve, if appropriate and necessary, using a #10 sieve. Wet samples may be difficult to homogenize and most projects require that samples as received be

used for analysis. Correction for percent solids is used to correct for moisture present in samples. With a laboratory spatula, place 1.25-g (±0.6-g) of each sample into separate, labeled sample cups. Check with Project Manager and/or project-specific QAPP (if available) about procedures that can be used for weighing out representative samples. Take small aliquots from different parts of the sample container to make up a representative1.25-g aliquot that is used for analysis. Record the weight of each sample in the sample preparation logbook to three significant places.

9.2 A method blank (MB), lab control sample (LCS), lab control sample duplicate (LCSD), matrix spike (MS), and matrix spike duplicate (MSD) should be prepared along with every batch of samples or at a rate of at least 5%. Samples used for MS/MSD should be homogenized to the extent possible to ensure homogeneity for the triplicate measurement on the sample. See individual determinative analytical methods and project specific quality assurance project plans (QAPP) for specific quality control requirements.

9.3 After weighing, place cups into the cup holders and then transfer them to the hotblock in the metals sample preparation hood.

9.4 Begin the digestion process by adding the current soil digestion spike solution to the sample(s) selected using a calibrated eppendorf pipette set to the correct volume.

9.5 ***Hard Digestion Procedure***: Samples can be prepared either for analysis on FLAA/ICP-AES or GFAA/ICP-MS. The extracts prepared for these two procedures are not interchangeable due to the presence of hydrochloric acid in the procedure used for FLAA/ICP-AES. Steps 9.5.1 to 9.5.5 are common to both procedures.

9.5.1 Add 10.0-mL of dilute nitric acid (1:1 Type-1 Water: conc.HNO_3) to each sample including method blank and the spiked QC samples.

9.5.2 Cover each cup with a watchglass and begin hotblock digestion at 95° C. Temperature of the hotblock must be verified each day prior to use with a thermometer immersed in sample beaker containing reference oil. Record beginning temperature in sample preparation logbook. Sample cups should be placed evenly in hotblock and allowed to reflux without boiling for 15-minutes. Remove from hotblock and allow samples to cool.

9.5.3 Add 5.0-mL of concentrated HNO_3, replace the cover, and reflux all samples for 30 minutes. NOTE: If red or brown fumes appear, continue with the addition of 5.0-mL concentrated nitric acid and refluxing for 30 minutes until no more brown fumes are given off by the sample to ensure complete oxidation. Using a ribbed watch glass allow the solution to evaporate to approximately 5-mL without boiling or heat at 95°C ± 5°C without boiling for two hours. Maintain a covering of solution over the bottom of the vessel at all times.

9.5.4 After the second (or more) reflux has finished and the samples have cooled, add 2-mL of Type-1 water and 3.0-mL of 30% hydrogen peroxide (H_2O_2, kept in the nearby standards/reagents refrigerator). Return cups to the hotblock to warm samples, causing an effervescing (not boiling) reaction to take place. **NOTE:** Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and

cool the vessels by removing them from the hotblock.

9.5.5 Add additional 30% H_2O_2 in approximately 1.0-mL aliquots and return samples to the hotblock, allowing the samples to effervesce until bubbling subsides (30 min.). Cool between steps. If samples are still effervescing, continue adding 30% hydrogen peroxide in approximately 1.0-mL aliquots until no more than 10-mL of 30% H_2O_2 has been added. Cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate until the volume has been reduced to approximately 5 mL or heat at 95°C ± 5°C without boiling for two hours. Maintain a covering of solution over the bottom of the vessel at all times. If samples are not effervescing, go to the next step. For FLAA/ICP-AES analysis, the next step is Section 9.5.6. For GFAA/ICP-MS analysis, go to Section 9.5.10.

9.5.6 ***For FLAA/ICP-AES analysis***, add 5.0-mL of concentrated HCl after samples have cooled.

9.5.7 Place samples back on the hotblock and reflux samples at 95°C ± 5°C without boiling for 15 minutes. Remove samples after 15 minutes and record final hotblock temperature in the sample preparation logbook.

9.5.8 After samples have cooled, rinse the cup walls with Type I water and bring the final volume to the 50-ml graduation with Type I water. Cap and shake the sample digestate and allow them to stand for approximately 30-mintes so that the particulates can settle down.

9.5.9 Insert an Environmental Express filter cartridge into the sample tubes to separate the soil from the digestate. (NOTE: For some USACE samples, centrifuging may be necessary instead of the separation procedure described above. If it is required, place tubes in centrifuge for 15 minutes to spin down sediment and heavier particulates.) Sample digestates are now ready for analysis.

9.5.10 ***For GFAA/ICP-MS analysis***, wait until samples have finished effervescing. Remove watchglass from cups to allow for evaporation and continue to heat samples for 15-minutes. Remove samples from hotblock and record final hotblock temperature in the sample preparation logbook. Continue with final volume adjustment and particulate removal to prepare digestates for analysis.

9.5.11 See Sections 9.5.8 and 9.5.9 above.

9.6 ***Soft Digestion Procedure:*** This procedure may be used to prepare samples for target analyte list (TAL) metals analysis, which includes analysis for antimony. Samples prepared using this procedure may be used for analysis on FLAA/ICP-AES. Analysis on GFAA/ICP-MS will require additional dilution to reduce the hydrochloric acid content. If concentrations of the elements in the final digestates (LCS/MS) are significantly different for the two procedures after appropriate dilution of the digestates, a duplicate set of QC samples may be prepared if analyses are required using both types of instruments.

9.6.1 Perform steps shown in Sections 9.1 to 9.4.

9.6.2 Add 2.5-mL of conc. HNO_3 and 10.0-mL of conc. HCL to each sample including method blank and the spiked QC samples.

9.6.3 Cover each cup with a watchglass and begin hotblock digestion at 95° C.

Temperature of the hotblock must be verified each day prior to use with a thermometer immersed in sample cup containing reference oil. Record beginning temperature in sample preparation logbook. Sample cups should be placed evenly on hotblocks and allowed to reflux for 15-minutes. Remove from hotblock and allow samples to cool.

9.6.4 Continue with final volume adjustment and particulate removal to prepare digestates for analysis as described in Sections 9.5.8 and 9.5.9 above.

9.7 ***PBM Digestion Procedure:*** This performance based (adapted from EPA Region-VII SOPs) method (PBM) may be used to prepare samples for target analyte list (TAL) metals analysis when excellent recoveries are required for antimony and silver. Silver recoveries are generally low for digestions performed using 3050-Hard and 3050-Soft digestion procedures. Samples prepared using this procedure may be used for analysis on FLAA/ICP-AES. Analysis on GFAA/ICP-MS will require additional dilution to reduce the hydrochloric acid content, which is three times greater than the amount used for 3050-Soft procedure.

9.7.1 Perform steps shown in Sections 9.1 to 9.4.

9.7.2 Add 5-mL of Aqua Regia (See Section 5.4) to each sample including method blank and the spiked QC samples.

9.7.3 Perform procedures as described in Section 9.6.3.

9.7.4 Perform procedures as described in Sections 9.5.8 and 9.5.9.

10.0 REFERENCES

10.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 6010B, Inductively Coupled Plasma Atomic Emission Spectrometry, December 1996.

10.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 6010C, Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 3, November 2000.

10.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 6020A, Inductively Coupled Plasma-Mass Spectrometry, January 1998.

10.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 7000A, Atomic Absorption Methods, July 1992.

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10.16 EPA Contract Laboratory Program SOW ILM05.0, Exhibit D.

10.17 USEPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, ILM04.0, April 1994.

10.18 10.13 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

10.19 AML Standard Operating Procedures, Q-Water-RC.

10.20 AML Standard Operating Procedures, Q-Subsampling-RC.

10.21 AML Standard Operating Procedures, Q-WasteMgmt-RD.

10.22 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

10.23 AML Standard Operating Procedures, Q-Controlcharts-RC.

Table 1

SAMPLE HOLDING TIMES, DIGESTED VOLUMES AND COLLECTION VOLUMES FOR METALS DETERMINATIONS IN AQUEOUS AND SOLID SAMPLES

Measurement	Digestion Amount	Sample Collection Volume or Weight	Holding Time Preservation (4°C)
Total	50-mL	600-mL	6 months, HNO3 to pH <2
Dissolved	50-mL	600-mL	6 months, filter on site; HNO3 to pH <2
Suspended	50-mL	600-mL	6 months, filter on site
Solid (Total)	1.25-g	200-gm	6 months

Appendix 1

Sample Preparation Logs

Analytical Management Laboratories, Inc. - Olathe, KS

ICP-MS, ICP-AES, GFAA, and FLAA SOIL - METALS Digestion Log

MDIG Batch # :		LCS & MS Spike Solution Names :	ICPMS-SS & ICPMS-AgS
META Batch # :		LCS & MS Spike Solution Reference #'s :	
Digestion Method :		LCS & MS Volumes :	
Digestion Date :		Spike Witness :	
Analyst Initials :		Reviewed By :	HNO3 Lot # :
Time Start :		HCl Lot # :	
Time Stop :		Temperature :	

CLIENT SAMPLE NUMBER	LABORATORY SAMPLE NUMBER	Sample Weight Digested (g)	Digestate Final Volume (mL)	% Solids	REMARKS
NA	MB	1.25	(50)	(100)	
NA	LCS	1.25	(50)	(100)	
NA	LCSD	1.25	(50)	(100)	

STANDARD OPERATING PROCEDURES

No. I-3060APrep-RC

ALKALINE DIGESTION OF SOIL, SEDIMENTS AND SLUDGES FOR HEXAVALENT CHROMIUM ANALYSIS BY MANUAL SPECTROPHOTOMETRY

October 28, 2005

APPROVED:

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ALKALINE DIGESTION OF SEDIMENTS, SLUDGES, AND SOILS FOR HEXAVALENT CHROMIUM ANALYSIS

1.0 SCOPE AND APPLICATION

This standard operating procedure is based on SW-846 Method 3060A, an alkaline digestion procedure for extracting hexavalent chromium [Cr (VI)] from soluble, adsorbed, and precipitated forms of chromium compounds in soils, sludges, sediments, and similar waste materials. Under the alkaline conditions of the extraction with added Mg^{2+} in a phosphate buffer, minimal reduction of Cr (VI) or oxidation of native Cr (III) occurs. Method-induced oxidation is usually not observed except in soils high in Mn and amended with soluble Cr (III) salts or freshly precipitated Cr $(OH)_3$. The quantification of Cr(VI) in alkaline digests is performed using AML SOPs which are based on SW-846 Method 7196 or similar colorimetric procedures. This SOP is not applicable to determining barium chromate in soil matrices.

2.0 SUMMARY OF METHOD

The sample is digested using 0.28M Na_2CO_3/0.5M NaOH solution and heating at 90-95□C for 60 minutes to dissolve the Cr(VI) and stabilize it against reduction to Cr(III). The pH of the digestate is carefully adjusted during the digestion procedure. The Cr(VI) reaction with diphenylcarbazide is used for the analysis of Cr(VI) solubilized in the alkaline digestate.

3.0 INTERFERENCES

The reducing/oxidizing tendency (as characterized by the determination of pH, ferrous iron, sulfides, ORP, TOC, COD, BOD and other related parameters) of the sample matrix will affect the tendency of Cr(VI) to exist or not exist in the unspiked sample(s) and their determination will assist in the interpretation of QC data for matrix spike recoveries outside conventionally accepted criteria. Certain substances, not typically found in the alkaline digests of soils, may interfere in the analytical methods for Cr(VI) following alkaline extraction if the concentrations of these interfering substances are high and the Cr(VI) concentration is low. For waste materials or soils containing soluble Cr(III) concentrations greater than four times the laboratory Cr(VI) reporting limit, Cr(VI) results obtained using this method may be biased high due to method-induced oxidation.

4.0 APPARATUS AND MATERIALS

Digestion vessel: borosilicate glass or quartz with a volume of 250 mL.
Graduated Cylinder: 100-mL or equivalent.
Volumetric Flasks: Class A glassware, 1000-mL and 100-mL, with stoppers or equivalent.
Vacuum Filtration Apparatus.
Cellulose or polycarbonate filter membrane (0.45 □m)
Heating and stirring device - capable of maintaining the digestion solution at 90-95□C.
Volumetric pipettes: Class A glassware, assorted sizes, as necessary.
Calibrated pH meter.

Calibrated balance.
Thermometer with NIST traceable calibration capable of measuring up to 100□C.

5.0 REAGENTS

Reagent Water free of interferences.

Nitric acid: 5.0 M HNO_3, analytical reagent grade. Do not use concentrated HNO_3 with an yellow tinge (contains nitrite, which reduces Cr(VI)) to make up 5.0 M solution. Store at room temperature (RT) in the dark.

Sodium carbonate (Na_2CO_3), anhydrous, analytical reagent grade. Store at RT.

Sodium hydroxide (NaOH), analytical reagent grade. Store at RT.

Magnesium Chloride ($MgCl_2$), anhydrous, analytical reagent grade. Store at RT.

Phosphate Buffer (1.0M) at pH 7: Dissolve 87.09 analytical reagent grade K_2HPO_4 and 68.04 g KH_2PO_4 into 700-mL of reagent water. Transfer to a 1L volumetric flask and dilute to volume.

Lead Chromate($PbCrO_4$), analytical reagent grade. The insoluble matrix spike is prepared by adding 10-20 mg of $PbCrO_4$ to a separate sample aliquot. Store under dry conditions at RT.

Digestion solution: Dissolve 20.0 ± 0.05 g NaOH and 30.0 ± 0.05 g Na_2CO_3 in reagent water in a one-liter volumetric flask and dilute to the mark. Store the solution in a tightly capped polyethylene bottle at RT. Prepare fresh monthly. Check the pH of the digestion solution (must be >11.5) before using. If not, discard.

Potassium dichromate ($K_2Cr_2O_7$) spiking solution (1000 mg/L Cr(VI)): Dissolve 2.829 g of dried (105°C) $K_2Cr_2O_7$ in reagent water in a one-liter volumetric flask and dilute to the mark. Alternatively, a 1000 mg/L Cr(VI) certified primary standard solution can be used (Fisher AAS standard or equivalent). Store at RT in a tightly sealed container for use up to six months.

Matrix spiking solution (100 mg/L Cr(VI)): Add 10.0 mL of the 1000 mg Cr(VI)/L made from $K_2Cr_2O_7$ spiking solution (Section 5.8) to a 100 mL volumetric flask and dilute to volume with reagent water. Mix well.

6.0. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of the SW-846 manual. Samples should be collected using devices and placed in containers that do not contain stainless steel (e.g., plastic or glass). Samples should be stored field-moist at 4 ± 2°C until analysis. Hexavalent chromium has been shown to be quantitatively stable in field-moist soil samples for 30 days from sample collection. In addition, Cr(VI) has also been shown to be stable in the alkaline digestate for up to 168 hours after extraction from soil. Treat all Cr(VI) waste materials with ascorbic acid or other reducing agent to reduce the Cr(VI) to Cr(III).

7.0 PROCEDURE

Adjust the temperature setting of each heating device used in the alkaline digestion to 90-95°C. Place 2.5 ± 0.10 g of the field-moist sample into a clean and labeled 250- mL beaker. Add 50-mL ± 1 mL of digestion solution to each sample using a graduated cylinder, and also add approximately 400 mg of $MgCl_2$ and 0.5 mL of 1.0M phosphate buffer. Cover all samples with watch glasses. Stir the samples continuously (unheated) for at least five minutes using an appropriate stirring device. Heat the samples to 90-95°C and maintain at that temperature for at least 60 minutes with continuous stirring. Gradually cool, with continued agitation, each solution to room temperature. Transfer the contents quantitatively to the filtration apparatus; rinsing the digestion vessel with 3 successive portions of reagent water. Transfer the rinsates to the filtration apparatus. Filter through a 0.45µ membrane filter. Rinse the inside of the filter flask and filter pad with reagent water and transfer the filtrate and the rinses to a clean 250-mL vessel. Store the filtered solid in a container at 4 ± 2°C.

Place an appropriate stirring device into the sample digest beaker, place the vessel on a stirrer, and, with constant stirring, slowly add 5.0-M nitric acid solution to the beaker dropwise. Adjust the pH of the solution to 7.5 ± 0.5 for Cr(VI) analysis using applicable AML SOP based on the SW-846 Colorimetric Method 7196A and monitor the pH with a pH meter. Do not overshoot while adjusting the pH to the desired pH range. If a flocculent precipitate should form, the sample should be either centrifuged or a filtered through a 0.45 µ pore membrane filter using larger pore prefilters if necessary. Transfer quantitatively the contents of the vessel to a 100-mL volumetric flask and adjust the sample volume to 100-mL with reagent water. Mix well. The sample digestates are now ready to be analyzed by the AML SOP based on SW-846 Method 7196A.

8.0 CALCULATIONS

Calculate sample concentration (µg/g or mg/kg) using the following equation:

$$\text{Concentration} = \frac{A * D * E}{B * C}$$

where:
- A = Concentration observed in the digest (µg/mL)
- B = Initial moist sample weight (g)
- C = % Solids/100
- D = Dilution Factor
- E = Final digest volume (mL)

Calculate RPDs for duplicates and spike recovery using standard procedures.

9.0 QUALITY CONTROL

For each sample digestion batch perform the following quality control analyses. Sample preparation procedures described below should be coordinated with sample analysis procedures

(spectrophotometry). Good coordination between the Analyst and Technician is essential for generating accurate Cr-VI data:

9.1 A preparation or method blank. The Cr(VI) level in the blank should be < MDL or 1/10 the regulatory limit or action level.

9.2 Laboratory Control Sample (LCS). Use the matrix spike solution to spike into 50 mL of digestion solution. Recovery must be within 80% to 120%.

9.3 A duplicate soil sample. RPD for duplicate samples must be ≤ 20%, if both the original and the duplicate are ≥ four times the laboratory reporting limit. A control limit of ± the laboratory reporting limit is used when either the original or the duplicate sample is < four times the laboratory reporting limit. Do not reject sample data if this is not attained since sample may be heterogeneous and varying levels of interference may be present.

9.4 One soluble and one insoluble pre-digestion matrix spike at a frequency of one each per batch of ≤ 20 field samples.

9.4.1 The soluble matrix spike sample is spiked with 1.0-mL of the spiking solution (equivalent to 40 mg Cr(VI)/Kg).

9.4.2 The insoluble matrix spike is prepared by adding 10-20 mg of $PbCrO_4$ to a separate sample aliquot. It is used to evaluate the dissolution during the digestion process.

9.4.3 Both matrix spikes are then carried through the digestion process.

9.4.4 Recommended acceptance range for matrix spike recoveries is 75-125%. If the matrix spike recoveries are not within these recovery limits, but the LCS recovery is within criteria, contact the client.

9.4.5 The Cr(VI) data may be valid for use despite the perceived "QC failure". It is the responsibility of the outside client to order the analysis of additional parameters and reanalysis of samples and/or the analysis of digested solids at additional cost as described in Section 3.0 of this SOP and sections 8.5.1 and 8.5.2 of the original SW method.

9.5 A post-digestion Cr(VI) matrix spike. The post-digestion matrix spike concentration should be equivalent to 40 mg/kg. Dilute the sample aliquot to a minimum extent, if necessary, so that the absorbance reading for both the unspiked sample aliquot and spiked aliquot are within the initial calibration curve. A guideline for the post-digestion matrix spike recovery is 85-115%. If not achieved, consider the Method of Standard Additions (MSA). If no spike is observed from the MSA, these results indicate that the matrix is incompatible with Cr(VI) and these digestates may contain soluble reducing agents for Cr(VI), such as fulvic acids. Document observations in a case narrative.

10.0REFERENCES

10.1Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual

Physical/Chemical Methods, SW-846, EPA, Method 6010B, Inductively Coupled Plasma Atomic Emission Spectrometry, December 1996.

10.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 6010C, Inductively Coupled Plasma Atomic Emission Spectrometry, Revision 3, November 2000.

10.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 6020A, Inductively Coupled Plasma-Mass Spectrometry, January 1998.

10.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 7000A, Atomic Absorption Methods, July 1992.

10.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 7000B, Flame Atomic Absorption Spectrophotometry, Draft Update IVA.

10.6 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 7010, Furnace Atomic Absorption Spectrophotometry, Revision 0, January 1998.

10.7 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Alkaline Digestion for Hexavalent Chromium, Method 3060A, Revision 1, December 1996.

10.8 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chromium, Hexavalent, Colorimetric, Method 7196A, Revision 1, July 1992.

10.9 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 3.2, Sample Preparation Methods, December 1996.

10.10 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 3.3, Methods for Determination of Inorganic Analytes, December 1996.

10.11 Methods for the Chemical Analysis of Water and Wastes, US EPA, EPA-600/4-79-020, Inductively Coupled Plasma, Method 200.7.

10.12 Methods for the Chemical Analysis of Water and Wastes, US EPA, EPA-600/4-79-020, AA Furnace Methods for Antimony (204.2), Arsenic (206.2), Cadmium (213.2), Chromium (218.2), Lead (239.2), Selenium (270.2), and Thallium (279.2).

10.13 Methods for the Determination of Metals in Environmental Samples, US EPA, EPA-600/4-91-01020, June 1991, Method 200.7, Determination of Metals and Trace Elements in Water and Wastes by Inductively Couples Plasma-Atomic Emission Spectrometry, p-31.

10.14 Methods for the Determination of Metals in Environmental Samples, US EPA, EPA-600/4-91-01020, June 1991, Method 200.8, Determination of Trace Elements in Water and Wastes by Inductively Couples Plasma-Mass Spectrometry, p-83.

10.15 Methods for the Determination of Metals in Environmental Samples, US EPA, EPA-600/4-91-01020, June 1991, Method 200.9, Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption Spectrometry, p-123.

10.16 EPA Contract Laboratory Program SOW ILM05.0, Exhibit D.

10.17 USEPA Contract Laboratory Program, Statement of Work for Inorganic Analysis,

ILM04.0, April 1994.

10.18 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

10.19 AML Standard Operating Procedures, Q-Water-RC.

10.20 AML Standard Operating Procedures, Q-Subsampling-RC.

10.21 AML Standard Operating Procedures, Q-WasteMgmt-RD.

10.22 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

10.23 AML Standard Operating Procedures, Q-Controlcharts-RC.

Table 1

SAMPLE HOLDING TIMES, DIGESTED VOLUMES AND COLLECTION VOLUMES FOR METALS DETERMINATIONS IN AQUEOUS AND SOLID SAMPLES

Measurement	Digestion Amount	Sample Collection Volume or Weight	Holding Time Preservation (4°C)
Total	50-mL	600-mL	6 months, HNO3 to pH <2
Dissolved	50-mL	600-mL	6 months, filter on site; HNO3 to pH <2
Suspended	50-mL	600-mL	6 months, filter on site
Solid (Total)	2.5-g	200-gm	6 months

Appendix 1

Sample Preparation Logs

Analytical Management Laboratories, Inc. - Olathe, KS

Alkaline Digestion Log

MDIG Batch # :		LCS & MS Spike Solution Names :	
META Batch # :		LCS & MS Spike Solution Reference #'s :	
Digestion Method :		LCS & MS Volumes :	
Digestion Date :		Spike Witness :	
Analyst Initials :		Reviewed By :	HNO3 Lot # :
Time Start :		HCl Lot # :	
Time Stop :		Temperature :	

CLIENT SAMPLE NUMBER	LABORATORY SAMPLE NUMBER	Sample Weight Digested (g)	Digestate Final Volume (mL)	% Solids	REMARKS
NA	MB	2.5	(100)	(100)	
NA	LCS	2.5	(100)	(100)	
NA	LCSD	2.5	(100)	(100)	

STANDARD OPERATING PROCEDURES

No. I-ICP-AES-Metals-RD

Metals Analysis by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) (EPA 200.7 and SW846 6010C)

October 21, 2005

APPROVED:

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1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) is based on EPA 200.7 and SW-846 Method 6010B/6010C (Draft Update IVB). This SOP is used to determine trace elements, including metals, in acid solution using inductively coupled plasma (ICP) atomic emission spectroscopy (AES).

1.2 This ICP-AES method is applicable to all elements listed in Table 1. Recommended analytical wavelengths, estimated MDLs and MQLs are shown for each element.

1.3 Detection limits, sensitivity, and optimum ranges of metals vary with the matrices. Method detection limits (MDLs) and method quantitation limits (MQLs) achievable at AML are shown in Tables 6 and 7 for water and soil respectively.

1.4 Samples consisting of various matrices require digestion prior to analysis. All sample processing steps of the analytical method should be included in the determination of method detection limits.

1.5 Groundwater samples that have been pre-filtered and acidified as well as drinking water samples that have been acidified may not require additional acid digestion prior to instrumental analysis. However, matrix-matching with the standards and procedures that incorporate use of internal standards for instrumental analysis are recommended. Calculation of analyte concentrations for non-digested samples should also take dilution factors into consideration.

1.6 Analysts performing this method should have at least one-year prior experience in correction of elemental, chemical, and physical interference.

2.0 SUMMARY OF METHOD

2.1 Samples are solubilized or digested prior to analysis by using one the methods listed in this SOP, including filtered water samples for dissolved elements.

2.2 This SOP utilizes a simultaneous ICP for element determination. The method measures element emitted light at various wavelengths. The nebulizer creates a sample aerosol, which is transported to the RF-argon plasma torch. The emitted light is dispersed by a grating and detected by a photo multiplier tube.

2.3 Some of the elements such as aluminum, iron, etc. emit several wavelengths of light, which may result in interference for other elements such as selenium and arsenic. Therefore, an inter-element corrections (IECs) procedure is performed, which allows the analyst to account for these potential interferences.

2.4 Other background correction techniques such as blank correction are also performed during the equipment standardization procedures.

3 INTERFERENCES

3.1 Interference from elements either having similar lambda maximum wavelength, or having secondary wavelength, which can coincide with the element of interest, is a major source of interference. Table 2 contains an example list of some of these potential interfering contributions.

3.2 To account for these potential interferences, an inter-element correction factor study is performed every six months. The instrument operator is responsible for performing these IEC studies and subsequently entering the correction factors in the methods used for analysis. In addition, IEC checks are performed every eight hours, or at the beginning and end of each run (whichever is more frequent), to verify that IEC correction factors are working. Sample analysis should not begin unless the ICSA and ICSAB standards checks have been performed within acceptance limits after instrument standardization.

3.3 Memory interference or "carryover" - Interfering contamination may occur when low levels of a target element are found immediately following the analysis of a sample containing high levels of a target element. An instrument blank is analyzed immediately following high samples. If target elements are found at or above detection limit in the instrument blank, the auto sampler and peristaltic pump lines are rinsed with 10 percent acid solution, followed by instrument blank rinse and reanalysis. No unknown sample analyses can proceed until corrective actions yield satisfactory results.

3.4 Evaporation of digested samples may lead to elevated reporting of element concentration. Digested samples are stored in 50-mL plastic bottles with snap-cap lids, at room temperature. Sample storage bottles are not recycled to prevent cross contamination.

3.5 Refer to Section 4 of SW-846 Method 6010C for a detailed discussion of the interferences.

4 APPARATUS AND MATERIALS

4.1 The current instrument used is a vacuum Thermo-Jerrell-Ash simultaneous Inductively Coupled Plasma model 61E with 23 element channels and one line for mercury peak profiling. The instrument is controlled by an IBM compatible computer using the current Thermo-Spec operating software, which contains automated procedures for background correction and IEC determinations. The computer is capable of storing several hundred data files. The instrument is under a full service contract with the manufacturer, which includes all non-disposable parts and labor.

4.2 The service contract includes 1 to 2 preventative maintenance visits per year, or when necessary. Instrument detection limit and method detection limit studies are performed once every six months, or when major maintenance's are performed, such as wavelength re-profile, element card replacement, photo-multiplier replacement, radio frequency overhaul,

optical replacement or cleanup. All major maintenance records are logged in the instrument maintenance logbook.

4.3 MDL studies are performed on each wavelength used, if an alternate wavelength is used, then MDLs are determined for the new wavelength. Inter-element correction studies are performed once every 6 months. Each sample analysis including standards, blanks, field samples, spikes, interference checks are recorded in an instrument run-log book.

4.4 The ICP has a TJA model AS300 auto sampler with a 319-position sample tray.

4.5 Liquid argon gas supply, high purity grade.

4.6 Class-A volumetric flasks - to mix and dilute standards to volume.

4.7 Auto sampler tubes, made of HDPE plastic - Purchase new. Do not recycle to prevent cross-contamination.

4.8 Peristaltic pump tubing (PVC) - Replace at least once a week.

4.9 Plasma torch - Clean once every 90 days, by soaking it with torch turned upside down in a concentrated aqua regia for 2 hours, followed by water rinse.

4.10 Transfer pipettes – for dilutions

4.11 Eppendorff Pipettes, 10 to 100-µL and 100 to 1000-µL volumes

4.12 Pipette Tips, disposable

5.0 **REAGENTS**

5.1 All chemicals used are reagent grade or better. If contamination is found at or above method detection limit all suspected chemicals are screened individually and disposed of safely.

5.2 Reagent water - Type-1 water is produced at the laboratory. See AML SOP on reagent water for details.

5.3 Acids – Trace Metals Grade

5.3.1 Trace metals grade concentrated Nitric Acid is purchased from Fisher Scientific or equivalent sources.

5.3.2 Trace metals grade concentrated Hydrochloric Acid is purchased from Fisher Scientific or equivalent sources.

6.0 **STANDARDS**

6.1 Individual and mixed metal standards for metals are purchased from SPEX, Ultra Scientific, Solutions Plus, Environmental Express, and other equivalent sources in concentration ranging from 100-ug/mL to 10000-ug/mL. Sources and concentrations are subject to change without notice.

6.2 Individual metal standards may be prepared from neat elements (>99.999% pure) purchased from reputed vendors (Alfa, etc.). Standard solutions may be prepared using detailed procedures that are included in Section 7.0 of SW-846 Method 6010C.

6.3 Stock Calibration Standard Solutions:

6.3.1 Stock Initial Calibration Standards. Five stock calibration standard solutions are obtained from Solutions Plus as described below:

6.3.1.1 AMSTD1 containing Beryllium, Cadmium, Lead, Manganese, Selenium, and Zinc at (100-ug/mL).

6.3.1.2 AMSTD2 containing Arsenic and Molybdenum at (100-ug/mL).

6.3.1.3 AMSTD3 containing Aluminum, Calcium, Potassium, and Sodium at (1000-ug/mL), and Chromium and Nickel at (100-ug/mL each).

6.3.1.4 AMSTD4 containing Magnesium at (1000-ug/mL), and Antimony, Silver, and Thallium at (100-ug/mL each).

6.3.1.5 AMSTD5 containing Iron at (1000-ug/mL), and Barium, Cobalt, Copper and Vanadium at (100-ug/mL).

6.3.2 Initial Calibration Standards. Dilute 10-mL of each mix described in Section 6.3.1 to 100-mL with 10-mL HCl, 5-mL HNO_3, and reagent water to volume. Label these solutions as STD-1 through STD-5. Use these solutions (see Appendix-1 for details) for standardizing the instrument.

6.4 Calibration Verification Standards. These second source standards are used to verify the calibration throughout the analytical run sequence.

6.4.1 The initial (ICV) and continuing (CCV) calibration verification standards are prepared by mixing and diluting five stock solutions (See Sections 6.3.1.1 to 6.3.1.5 for concentrations) from a different vendor to produce mixed metal standards. These stock solutions are labeled as AM Standard 1 through AM Standard 5. The concentrations of the analytes in these solutions are shown in Table 3 (see Appendix 2 for details on their preparation).

6.4.2 The CCV contains elements at one half of the concentration of the elements in the ICV standard.

6.4.3 The ICV standard may also be obtained as a single mix from a second source vendor at a higher concentration and used after a simple dilution (e.g. 1:10). The CCV should be prepared by 1:2 dilution of the ICV standard.

6.4.4 CRI or MQL Check Standard – This standard is prepared at a concentration close to two times the EPA contract laboratory program (CLP) required detection limit (CRDL). The concentration levels in the CRI solution is adjusted to yield observed values that are within 20% of the true values during routine analysis. The concentration of the analytes in the CRI solution equals the method quantitation limits (MQLs) for each analyte. The CRI solution is prepared from a simple dilution of individual element standards (see Appendix 2 for details). The concentrations of the analytes in the CRI solution are shown in Table 3

6.5 Standards for the determination of linear range. Appropriate dilutions of the individual metal standards in 1% HNO_3 (diluent used) are used to prepare a series of standard solutions of varying concentrations to determine the instrument linear range. Small quantities (10-mL) of these solutions are prepared (see Appendix 3 for details) to minimize disposal problems. The results of the linear range study are shown in

Table 4. Linear range studies should be performed annually. Verification of the linear range may be performed more frequently (e.g. quarterly) to support specific analytical programs.

6.6 High Level Calibration Verification Standard. This standard solution (HLSTD) is used to provide daily demonstration of linearity for specific analytes at concentrations that are greater than those in the initial calibration standard (used for instrument standardization and read-back). The HLSTD contains potentially toxic metals (Ba at 20-mg/L, Cr at 50-mg/L, Cu at 50-mg/L, Pb at 100-mg/L, Mn at 50-mg/L, Ni at 50-mg/L, and Zn at 50-mg/L) that may be present in sample digestates at levels greater than in the standard used for instrument calibration. Analysis of the HLSTD provides daily verification of the instrument linear range (Section 6.4). Checking instrument calibration by analyzing this standard is intended to minimize the number of dilutions that may be needed to support specific programs (e.g. USACE). The HLSTD is prepared by dilution of the 10,000-mg/L individual element standards (see Appendix 2 for details).

6.7 Interference Check Samples - Stock solutions to prepare various interference check samples are purchased from Solutions Plus. They include custom mix solutions (catalogue # INTF-A and AML-1 blend). Details of the preparation of the interference check standards are documented in analyst logbooks (see Appendix 4 for details).

6.7.1 ICSA: Dilute 10-mL of the ICSA mix (INTF-A) purchased from Solutions to 100-mL with 10-mL HCl, 5-mL HNO_3, and reagent water to volume.

6.7.2 ICSAB: Dilute 10-mL of ICSA mix and 1-mL of Solutions Plus AML-1 blend to 100-mL with 10-mL HCl, 5-mL of HNO_3, and reagent water to volume.

6.7.3 ICSA2: This standard is a specific check on the interferences caused by Manganese at 20-mg/L). Dilute 2-mL of Manganese (1000-ug/mL) to 100-mLs with 10-mLs HCl, 5-mLs of HNO_3 and de-ionized water.

6.7.4 ICSA3: This standard is a specific check on the interferences caused by Vanadium at (20-mg/L). Dilute 2-mL of Vanadium (1000-ug/mL) to 100-mLs with 10-mLs HCl, 5-mLs of HNO_3 and de-ionized water.

6.7.5 ICSAB2: This standard contains the target analytes as well as the Manganese and Vanadium interferents. Dilute 2-mL each of Manganese (1000-ug/mL) and Vanadium (1000 ug/mL) with 1-mL of Solutions Plus AML-1 blend to 100-mL with 10-mL HCl, 5-mL HNO3, and reagent water to volume.

Note: Additional (unusual) interferences (high Arsenic, Molybdenum, Thallium, etc.) should be addressed on a case by case basis as they arise. Contact the Team Leader for specific instructions.

6.8 Laboratory Control Standard/Matrix Spike: Two spike solutions are used for each matrix, soil and water. One of these solutions (ICPAgS) for each matrix contains

only silver. The other solutions contain a mixture of 22 elements at varying concentrations, which are different for water (ICPWS) and soil (ICPSS). Document directions for the preparation of the ICP spike solutions (see Appendix 5 for details) in logbooks.

6.9 Blanks: Calibration blank is used during initial standardization, while reagent blank is used to account for acid content variance.

6.9.1 The calibration blank has the same amount of acid as the element calibration standards.

6.9.2 The Method Blank is digested at the same acid level as the unknown samples and spike samples. Method blanks are analyzed as per QA/AC schedule listed in this Standard Operating Procedure or as per regulatory documents governing the batch analysis. The final volume of the unknown samples, spikes and method blanks must be the same.

6.10 Traceability:

6.10.1 All metals standards are to be logged into the Standards and Reagents logbook immediately after they are prepared. The standards are each given a unique reference number based on the date and sequence of preparation (e.g., if two standards were prepared on December 5th 1999, the reference #'s assigned to them would be M991205-01 and M991205-02.

6.10.2 All ICP Instrument standards are to be identified in the ThermoSPEC software auto-sampler tables by their reference number, book number, and page number. This is accomplished by:

6.10.2.1 From the ThermoSPEC main menu; Operation/Autosampler Setup press Enter.

6.10.2.2 Press F6=List to choose the applicable table, type in the table number, press Enter.

6.10.2.3 Press Enter again to access the table.

6.10.2.4 Press F1=Edit Set

6.10.2.5 Press F1=Ed Samples

6.10.2.6 Press Alt F2=EdSampInfo

6.10.2.7 Toggle (arrow key) right to the Comment column.

6.10.2.8 Press F1=Edit Mode

6.10.2.9 Type in the Standards trace-ability numbers (e.g., M991205-01, Book 1, Page 2)

6.10.2.10 Edit all Instrument Standards in the table accordingly.

6.10.2.11 Press F9=Done/Keep 4 times to save the table to file.

7.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

7.1 Definitions of terms:

7.1.1 Total Metals: The concentration of metals determined in a sample following digestion.

7.1.2 Suspended Metals: The concentration of metals determined in the portion of a sample that has been filtered through 2.0 um filters and followed by digestion.

7.1.3 Dissolved Metals: The concentration of metals determined in a sample after the sample is filtered through a 0.45 um filter.

7.1.4 Total recoverable metals: The concentration of metals in an unfiltered sample following treatment with hot dilute mineral acid.

7.2 See Table 5 for volume (or weight) of sample to be collected and volumes used for analysis.

7.3 Aqueous Samples - See project specific field sampling plan (FSP) for sample collection and preservation instructions. A liter or 500-mL plastic bottle with screw cap may be used for collecting aqueous samples in accordance with EPA SW-846 methods 6010B and 7000 series methods. Order pre-cleaned bottles containing the nitric acid preservative directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each water sample to be collected.

7.3.1 Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. Provide two additional containers for MS and MSD sample collection.

7.3.2 Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.

7.4 Soil Samples - A 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples in accordance with EPA SW-846 Method 6010B or 7000 series methods. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each solid sample.

7.4.1 Quality control samples such as MS and MSD should be treated as regular environmental samples. Provide two additional containers for MS and MSD sample collection.

7.4.2 Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.

7.5 These samples may be shared with samples collected for other analyses such as metals by AA (7000), chlorinated pesticides (8081A), PCBs (8082), other pesticides (8140, etc.), semivolatile organics (8270C) and diesel range organics (DRO, 8015-mod.) or TPH.

7.6 Samples must be extracted and analyzed within the specified holding times (180-days for elements except mercury) for the results to be considered reflective of total concentrations. Analytical data generated outside of the specified holding times must be considered to be minimum values only. Such data may be used to demonstrate that a waste is hazardous where it shows the concentration of a constituent to be above the regulatory threshold but cannot be used to demonstrate that a waste is not hazardous. The holding time for the extraction and/or analysis of aqueous and soil samples (including TCLP leachate preparations) is 6-months from the date of sample collection.

8.0 HEALTH AND SAFETY

8.1 Calibration standards and other standards used for quality control contain a number of toxic heavy metals at high concentrations. The toxicity of these complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

8.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

8.3 Reagents used such as acids are highly corrosive. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

8.4 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

8.5 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

9.0 WASTE DISPOSAL

9.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

9.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

9.3 Soil samples that have undergone extraction should be free of contaminants. They can be disposed off into sanitary dumpsters. Dispose of sample extracts containing high levels of acid in accordance with applicable AML SOPs.

10.0 PROCEDURE

Procedures described below may be supplemented with notes taken by the analyst during training. Actual steps to be followed by the analyst may differ from those described below due to the detailed nature of the procedure that follows.

10.1 Instrument Set-up

10.1.1 Establish liquid argon flow to the unit at approximately 80 psi. Check water level in the water cooler.

10.1.2 Check vacuum: Red set at 20, black at approximately 10.

10.1.3 On the ThermoSPEC software, go to Setup, then Plasma Control Panel. Press F1 then F9 to start the flame. After the Plasma is on, press Enter, then F2 to “Set Levels”. Set Aux Gas to *High*, Nebulizer to *on*, Nebulizer flow rate to *0.63*, and RPM to *100*. Press F9 to set the changes, then press Escape to return to the TermoSPEC main menu.

10.1.4 Wait one hour before beginning calibration procedures.

10.2 Instrument Operation. See supervisor for the specific run sequence to be used. Instructions for a typical run (see Appendix 6 for details) are shown below.

10.2.1 Update the datafile on the first working day of each month. On the ThermoSPEC software, go to Development/Methods press Enter; 6010 will come up by default - press Enter; press F3=Method Info; toggle (arrow key) down to Analysis Data File and type in the three alphabetical character name for the month (e.g. Jan for January) and the two digit numeric (01 for 2001) for the year (e.g., Jan01 for January 2001); press F9=Done/Keep twice to save to method file.

10.2.2 Perform Mercury Peak Profile (-2.00 ± 0.10), save to method file.

10.2.3 Perform initial calibration including blank standard using standards described in Section 6.4.1.

10.2.4 Read back calibration standard. Result should be within 5 % of true value. If not, recheck mercury alignment and re-calibrate.

10.2.5 Analyze the HLSTD. Results should be within 10% of the true values. If the results are not within 10% for any of the analytes appropriate dilutions of the digestates will be necessary. NOTE: This is an AML procedure performed in response to comments made by one of our clients (USACE).

10.2.6 Analyze ICV. Result should be within 10%. If not, re-analyze ICV. If values still not within 10%, check mercury alignment and re-calibrate. If values still not within 10%, obtain new standards and repeat procedure.

10.2.7 Analyze ICB. All values should be below blank criteria.

10.2.8 Analyze a CRDL standard (CRI) or the MQL/PQL standard as applicable.

10.2.9 Analyze ICSA. All interferent values should be within 20% of true value. If any target analytes indicate a bias, adjust the IEC's accordingly and reanalyze ICSA.

10.2.10 Analyze ICSAB. All interferent values should be within 20% of true value.

10.2.11 Analyze ICSA2 & ICSA3. All interferent values should be within 20% of true value. If any target analytes indicate a bias, adjust the IEC's accordingly and reanalyze ICSA's.

10.2.12 Analyze ICSAB2. All values should be within 20% of true value. NOTE: only after all above criteria is met, can sample analysis begin.

10.2.13 Analyze CCV. Results should be within 10% of true value. If not, correct the problem.

10.2.14 Analyze CCB.

10.2.15 Analyze the Method Blank. Method blank levels should not exceed one half of the applicable reporting limits (< 1/2 RL) for target analytes. It cannot exceed applicable reporting limits (<RL) for common contaminants.

10.2.16 Analyze LCS (and LCSD). Recoveries should be within in-house statistical control limits. See Section 12.10 for additional details.

10.2.17 Analyze additional 7 or 8 samples (total of 10 including Blank, LCS and LCSD, if present).

10.2.18 Analyze CCV. Results should be within 10% of true value. If not, all samples previous to the failing CCV, and following the last successful CCV, need to be reanalyzed.

10.2.19 Analyze CCB.

10.2.20 Analyze 10 samples, followed by CCV, CCB and continue this sequence until all samples are analyzed.

10.2.21 End sample run with CCV, CCB, CRI, ICSA, ICSAB, ICSA2, ICSA3, ICSAB2, CCV, and CCB.

10.3 Instrument Shut-Down

10.3.1 On the ThermoSPEC software, go to Setup, then Plasma Control Panel. Press F5 to turn plasma off.

10.3.2 Press F7 to shut down

10.3.3 Turn off Argon gas

10.3.4 Release pressure on the pump tubing

10.4 Analysis Sequence – Perform analysis of standards and samples using the sequence described above. Refer to Appendix 6 of this SOP for a run log, which shows the typical sequence that is employed for routine analysis.

11.0 QUALITY CONTROL

11.1 Quality control (QC) procedures are used to ensure that proper sample preparation techniques and instrument calibration procedures are performed. This involves instrument standardization, establishing the linear range for each target element, establishing instrument and method detection limits (IDL and MDL), initial verification of the calibration (ICV), continuing verifications of the calibration during the analysis sequence (CCVs), final verification of the calibration at the end of sample analysis (FCV), and the verification of calibration blanks throughout the analytical sequence (ICB, CCBs and FCB). It also involves an evaluation of the interferences using check samples (ICSA, ICSAB, etc.) and an evaluation of the practical or method quantitation limits (PQL or MQL) for each target analyte.

11.2 The QC procedures also involve the analysis of method blanks (MB), laboratory control samples (LCS and LCSD), and matrix spikes (MS and MSD) for each samples prepared as a quality control (QC) batch. Interference detection procedures such as serial dilution (SD), post digestion spikes (PDS), and method of standard addition (MSA), if necessary are also elements of the batch-specific QC program for this analysis.

11.3 Establish inter element correction factors, linear range, instrument detection limits (IDL), method detection limits (MDL, see Appendix 13 for details on MDL studies), lowest level for reporting or reporting limits (RL), and method quantitation limits (MQL) for each element of interest. See AML SOPs for more detailed information on these QC parameters.

11.3.1 Analyze a MDL check sample on a quarterly basis. This is a qualitative check that also establishes the lowest reporting limit.

11.3.2 According to SW-846 Method 6010C, the MDL check sample is spiked into reagent water at 2-3 times the MDL and is carried through the entire analytical procedure. Detection limits are verified when all analytes in the MDL check sample are detected.

11.3.3 Spiking the water at the lowest level for reporting (LLR) is recommended for conducting the MDL check sample studies.

11.4 Standardization Check - The solutions used for instrument standardization are also analyzed as unknown values. The observed values should be within 5% of the true values. This is an optional AML procedure to verify instrument stability. This procedure may be a requirement for some programs.

11.5 High Standard Check - The HLSTD solution is used to check linearity at levels that are greater than the solution used for instrument standardization but less than the linear range (see Table 4). The observed values should be within 10% of the true values. This is an optional AML procedure (recommended by USACE) to verify instrument stability. Analysis may be continued if one or metals do not meet criteria. Re-analysis using dilutions will be necessary to meet the requirements for some programs (e.g. USACE).

11.6 The practical or method quantitation limits (MQL) should be verified by running PQL check samples (CRI). This check should follow ICV analysis. The observed values should be within 20% of the true values. The concentration of individual analytes may be optimized to provide low MQLs, while assuring 20% accuracy at MQL.

11.7 ICV - Initial calibration verification (ICV) must be performed after instrument calibration procedures have been completed. The ICV should be within 10% of the true value. If reanalysis of the ICV is unsatisfactory, recalibration is required. If the problem persists the analysis is halted and instrument maintenance is performed.

11.8 CCV/FCV - The continuing calibration verification (CCV) and final calibration verification (FCV) check standard sample is analyzed every 10th sample and at the end of sequence respectively. They should be within 10% of the true value. Otherwise, data for the previous ten samples may be considered unusable (or estimated depending on the monitoring program) and must be reanalyzed.

11.9 ICB/CCB/FCB - An initial calibration blank (ICB) is analyzed to verify that no target analytes have been inadvertently introduced into the calibration reagents. ICB analysis must be performed after instrument calibration procedures have been completed. The continuing calibration blanks (CCBs) and final calibration blank (FCB) are analyzed every 10th sample and at the end of sequence respectively.

11.9.1 The calibration blanks follow the analysis of calibration verification in the run sequence.

11.9.2 The calibration blank results should be <MDL for the target analytes. Use criteria specified in project plans if they are different from the default criteria.

11.9.3 Reanalysis of the blanks and associated samples is necessary if blank criteria are not met. If reanalysis of the calibration blanks are

unsatisfactory, recalibration is required. If the problem persists the analysis is halted and the source of contamination determined.

11.10 Inter-element Correction Factors (IECs) must be verified during every eight hours, or at the beginning and end of each run (whichever is more frequent). This is performed by running ICSA and ICSAB standards after ICV, ICB and CRI standards initially, before every other CCV, CCB and CRI during the sequence and before the FCV and FCB at the end of sequence.

11.10.1 If the instrument produces negative or positive responses for corrected elements, the instrument is out of compliance and IEC factors must be recalculated, entered into the analytical method, and documented into the IEC Logbook.

11.10.2 The observed concentration of interfering elements should be within 10% of the true values. The observed value for target elements should be within 20% of the true values.

11.10.3 Elements not present in the solutions should meet blank criteria that are described elsewhere. If reanalysis of the ICSA and ICSAB are unsatisfactory, check mercury alignment, re-calibrate, obtain new standards, and reanalyze.

11.11 Laboratory Control Samples (LCS) - The frequency of analysis of LCS analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples. LCS can be run in duplicate to obtain precision (RPD) information.

11.11.1 The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. Normally, the LCS spike levels should be low, near action levels (when available) to demonstrate the ability of the laboratory to accurately analyze samples at that level. Other recommended spike levels include concentration ranges that are between the reporting limit and the mid range of the calibration. Since analytes for this method include geochemicals that are present in significant quantities in soil and groundwater, client requirements often include recommended LCS spike levels that are identical to the matrix spike levels, which should exceed expected matrix levels. Client specifications should supersede other requirements that are more theoretical in nature.

11.11.2 LCS recoveries should be within QC limits (statistical control limits, method specified limits, and DoD QSM limits). When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform accurate analysis in a clean matrix.

11.11.3 LCS can be run in duplicate to obtain precision (RPD) information. The RPD for duplicates, which includes LCS duplicates should generally be within method-specified QC limits (typically 25%) for MS or sample duplicates.

11.12 Matrix Spike (MS) Analysis - The frequency of analysis of MS analysis is equivalent to a minimum of 2 per 20 samples or 2 per batch if less than 20 samples.

11.12.1 The MS recoveries should be within QC limits (see Tables 6 and 7), which are generally set at ±25% of the spiked value (SW-846 Method 6010C criteria).

11.12.2 MS is run in duplicate to obtain precision (RPD) information. The RPD for duplicates, which includes MS duplicates should be within QC limits (see Tables 6 and 7), which are generally set at 20% for RPD (SW-846 Method 6010C criteria).

11.13 Method Blank (MB) Analysis - The frequency of analysis of MB analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples.

11.13.1 Report association of samples with MB (and LCS/LCSD, MS/MSD, and SD/PDS) on Form-4 equivalents (see Appendix 16 for details).

11.13.2 The MB should meet blank criteria (<LLR or <0.5*MQL).

11.14 Post Digestion Spike (PDS) and Serial Dilution Tests – The new SW-846 method revision (6010C) recommends performing the PDS test first on the sample used for MS/MSD analysis. If both the MS/MSD and the PDS recoveries fail, matrix effects are confirmed. If the serial dilution also fails, a chemical or physical interference effect should be suspected.

11.15 Post Digestion Spike Procedure or Recovery Test for Interference - Typically, sample spike level used at AML is set at the same level as LCS and MS spike levels for low concentration samples.

11.15.1 The spike addition should produce a minimum level of 10 times and a maximum of 100 times the MDL. If the spike levels are relatively low compared to the sample amounts, appropriate dilutions of the sample extract should be employed (typically in increments of 1:5) so that spike amounts are at least 5 times greater than the original sample concentration.

11.15.2 According to the older revision of the method (6010B), the spike recovery should be within ±25% of the true value (75% to 125% for spike recovery). This QC limit has been vacated.

11.15.3 The revised SW-846 Method (6010C) requires tighter limits (±20% of the true value) for PDS recoveries (80% to 120%).

11.15.4 If the sample concentrations are relatively high and PDS studies are not practical, perform serial dilution studies.

11.16 Serial Dilution (SD) Procedure – Perform 1:5 dilution of one sample from each analytical batch and compare the results for the two analyses. Samples selected for matrix spike analysis can be selected for routine performance of this test.

11.16.1 The analyte concentration should minimally be 10 times the MDL after 1:5 dilution for the SD results to be applicable.

11.16.2 If the SD study is valid, and the results for the SD sample agree within ±10% of the results for the original sample, the absence of interferences is indicated.

11.16.3 Perform serial dilution test on another sample, if the original (undiluted) value is less than 50 times the MDL.

11.16.4 Use of an alternate wavelength for the ICP-AES measurements, computerized compensation in cases of spectral overlap, comparison of the results from an alternate method (e.g. ICP-MS) are acceptable corrective action procedures. See Section 4.0 of Method 6010C for potential interference and recommended solutions.

11.17 Method of Standard Additions (MSA) – This relatively tedious procedure can be used when interferences are detected from dilution and recovery tests performed as described above. The simplest MSA procedure is described below:

11.17.1 A known volume of the sample extract (V_x) is spiked with a known volume (V_s) of a standard solution of known concentration (C_s) so that the spiked sample concentration (S_a) is approximately twice the concentration of the unspiked sample (S_b).

11.17.2 The unspiked sample is diluted with the same volume (V_s) of reagent water.

11.17.3 Both samples are analyzed on ICP-AES and the unknown sample concentration (C_x) is calculated using the following equation:

$$C_x = \frac{S_b * V_s * C_s}{(S_a - S_b) * V_x}$$

where
V_x = Volume of sample extract
V_s = Volume of standard solution spiked
S_a = Measured concentration of spiked sample
S_b = Measured concentration of the unspiked (diluted) sample
C_s = Concentration of the spiking standard
C_x = Concentration of the unknown sample to be reported

11.18 The MSA can also be performed using multiple spikes and the C_x calculated graphically by plotting the observed result against the spiked concentration on an Excel spreadsheet (see Appendix 7 for details). Use at least 3 spiking levels at approximately 50%, 100%, and 150% of the original concentration in addition to the analysis of the diluted sample. The spiked and unspiked samples should contain the same amount of sample extract diluted to the same final volume. This procedure may be performed for one sample in the QC batch, while performing the MSA procedure outlined in Section 11.16 for all other samples.

11.19 An alternative to using the MSA is to use an internal standard (e.g. Yttrium or Scandium) to overcome matrix interferences, especially in high solids matrices.

11.20 Any sample that has an element response above the established linearity of the instrument (Table 4) is diluted and reanalyzed. Samples containing high levels of elements such as Iron and Aluminum are carefully screened for false positives or

negative reporting. Confirmation of low level of elements such as Arsenic, Selenium, Thallium, Chromium, Lead and Cadmium must be made using Graphite Furnace Atomic Absorption (GFAA, SW-846 Method 7000) or ICP-Mass Spectrometry (ICP-MS, SW-846 Method 6020A) techniques. If any of these elements are present at levels greater than their respective reporting limits but less than the levels used for initial calibration (instrument standardization), the results from this analysis can be reported. If the sample digestates levels are higher but less than a valid HLSTD level, additional dilution and re-analysis of the sample digestates will not be necessary. NOTE: GFAA and ICP-MS techniques require unique digestion procedures as described in SW-846. HCl interferes with Thallium analysis producing false negative results.

11.21 The analysts may document sample and QC data on Excel spreadsheets for projects that do not require Level III or Level IV reporting (see Appendix 14 for details). Data contained in the spreadsheets may be used for review and the preparation of final reports for clients.

11.22 Whenever possible, the laboratory should analyze standard reference materials (e.g. from ERA or NIST) and participate in relevant performance evaluation studies.

12 **DATA VERIFICATION AND VALIDATION -** This section will provide guidance when evaluating and validating data generated by this SOP. It provides procedures to assess if data produced is valid and legally defensible. Document the review on checklists (see Appendix 8 for details) and/or on the case narrative as appropriate. Corrective action procedures are described below (see Appendix 9 for details).

12.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) projects may have additional requirements that are related to USACE laboratory validation (CX), USACE District requirements, government contract requirements, specific USACE project requirements or USACE Architect/Engineer (A/E) requirements. See supervisor for client specification (see Appendix 10 for more details on USACE, AFCEE, etc. clients). The USACE blank criteria shown in appendix 10 has been modified by USACE auditor(s) as one-half of the applicable MQL. The USACE requirements shown in appendix 10 and elsewhere in this SOP are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may be unattainable in routine use for sample analysis. Since USACE MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

12.2 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality control requirements. Data generated under these conditions may need to

be labeled as screening data. Exceptions and data qualifications necessary should be documented on the checklists and eventually on the case narrative.

12.3 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist, perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete. For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete. For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director will review the checklists (initial/date as necessary), prepare case narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

12.4 HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

12.4.1 WATER - The holding times for sample digestion and analysis is 180 days from sampling. Water samples include TCLP leachates.

12.4.2 SOIL – The holding times for sample digestion and analysis is 180 days from sampling.

12.4.3 WASTE - The holding times for TCLP leachate preparation is 180 days from sampling.

12.4.4 Holding times are established by comparing the chain of custody sampling date with the date of the injection.

12.4.5 ACTION – If holding times have been exceeded (an unlikely event for this analysis), contact the client.

12.4.6 ACTION - All positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier.

12.4.7 ACTION - If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or invalid (I).

12.5STANDARDIZATION CHECK AND INITIAL CALIBRATION VERIFICATION (ICAL and ICV) – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the instrument is capable of producing data within a range that is linear. ICV demonstrates that instrument performance has not changed since the generation of ICAL.

12.5.1 Standardization Check (read-back) – Verify that the true values for standard read-backs are within 5% of the true values. They should not deviate by more than 10%.

12.5.2 ACTION – This is an AML QC requirement. If analysis is performed in accordance with EPA CLP requirements, stop when the true values exceed 5% criteria. Correct the problem and repeat instrument standardization. Otherwise, use the 10% criteria or skip this procedure altogether.

12.6HLSTD - Check to see that HLSTD is within ± 10% of the true value. It is recommended that the RSD for multiple readings be within 5%.

12.6.1 ACTION - If QC criteria are not satisfied, inspect and correct the problem and reanalyze HLSTD for acceptable results. Since this is an AML QC requirement, sample analysis

may be continued. Sample digestates exceeding the levels used for instrument standardization may require re-analysis under dilution for specific elements. See Table 4 for the list of covered elements.

12.7 ICV – Check to see that ICV is within ± 10% of the true value and the %RSD for multiple readings are within 5%.

12.7.1 If QC criteria is not satisfied, correct the problem and reanalyze ICV (and ICAL calibration solutions, if necessary) for an acceptable ICV.

12.7.2 ACTION – Contact client if corrective action does not resolve problem within the schedule for the project for rapid turnaround projects. Positive results for compounds not meeting the criteria may need to be qualified as estimated (J) by the user (client). Report detects and non-detects without qualifiers and mention these outliners in the case narrative.

12.8 CONTINUING CALIBRATION (CCVs and FCV)– The continuing and final calibration verification solutions are used to check that the instrument is still operating under the linear range of the initial calibration and has remained stable over the entire analytical sequence.

12.8.1 CCVs and FCV values should not deviate more than ± 10% of the true value. The RSD for multiple readings should not exceed 5%.

12.8.2 ACTION – If QC criteria is not satisfied, correct the problem and reanalyze CCVs and FCV and all associated samples. Check associated sample results that are greater than MQL. Associates samples include all samples that precede and follow the affected CCV. Contact client if corrective action does not resolve problem within the schedule for the project. Positive results for compounds not meeting the criteria may be qualified as estimated (J) by the user (client). Report non-detects without qualifiers and mention these outliners in the case narrative.

12.9 METHOD BLANKS (MB) and CALIBRATION BLANKS (ICB, CCBs, and FCB) – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Sporadic cases of contamination may be difficult to control. However, daily contamination would not be acceptable. When the concentrations of any target analyte in the MB exceeds the applicable QC limit (one half of the MQL or one half of the project specific reporting limit), method blank is considered to be positive and steps shall be taken to find, reduce, and/or eliminate the source of this contamination. Additional project-specific corrective actions may be necessary. Qualify associated sample data to alert the user about method blank contamination in the laboratory. Sample data qualification by the client based on all the blank results (laboratory MB, calibration blanks, equipment blank, and other field blanks) is performed by the user (client). Criteria for blank is especially confusing due to the multitude of benchmarks (IDL, MDL, MQL) and a lack of coherent EPA policy. Analyst discretion and professional judgment are very important in generating acceptable data.

12.9.1 Method blanks and calibration blanks should be free of all target analytes and other interfering non-target analytes. The AML criteria is ±0.5*MQL (default) or one half of the reporting limit (project specific).

12.9.2 To be acceptable for some projects, blank results may not exceed the default criteria and/or one or more of the following criteria that may be applicable: ±3*IDL, ±MDL, etc. Apply appropriate project-specific criteria for sample analysis.

12.9.3 Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples. If levels greater than one half the MQLs are present, inspect and correct the problem. Reanalyze all associated samples. Contact client if other criteria are applicable.

12.9.4 Exceptions to the criteria may include quick turnaround time projects.

12.9.5 ACTION - If an analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary, other than discussing the situation in the case narrative.

12.9.6 ACTION – Any compound detected in the sample which were also detected in associated method blank at levels greater than one half the MQLs should be re-digested and re-analyzed unless other client criteria are applicable.

12.9.7 ACTION – All positive results for associated samples are reported with a "B" code, if contaminants are found in the method blank at levels greater than the reporting limit.

12.9.8 ACTION - If the sample amount is more than 10-times the method blank amount, re-extraction and re-analysis of the affected sample is not necessary.

12.9.9 ACTION - If the sample amounts are less than 10-times the blank level, reanalyze the method blank, and any samples containing the same contaminant. If the contamination remains, the contaminated samples of the batch should be re-prepared and reanalyzed with a new method blank and batch specific QC samples.

12.9.10 ACTION – If gross contamination is observed, all affected elements are qualified as invalid (I). Re-extraction and re-analysis will be necessary.

12.10 LABORATORY CONTROL STANDARD (LCS) – A LCS analysis is used to evaluate method competence in the absence of matrix effects. The LCS is evaluated by comparing the percent recovery for the target analytes to the QC limits (Tables 6 and 7). Default QC limits are based on limits proposed by the Department of Defense Quality Systems Manual (QSM, Final Version 2, June 2002). Other evaluation procedures may include comparison with those determined by project-specific DQOs (See project QAPP, if available) or laboratory statistical control limits. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives.

12.10.1 Document LCS/LCSD results in Form-1 equivalents and recoveries/RPD in Form-3 equivalents (see Appendix 11 for details). The %R and RPD should be within QC limits. Check and flag outliers with an asterisk (*).

12.10.2 ACTION - Initially, the effect of QC failure on the samples should be evaluated. Regardless of this assessment, steps shall be taken to find the source of the problem and correct it. The case narrative shall discuss the corrective action taken and any other information.

12.10.3 When multiple (>10) target analytes reported, the DoD QSM acceptance criteria may allow for the sporadic marginal failure or marginal exceedances ("ME") for a few target analytes. This allowance is made only for specific hard-to-analyze elements such as Mo, Se, and Ag in aqueous and soil samples. Allowances for additional elements (Al, Sb, and Zn) in soil samples are included in the QSM.

12.10.4 ACTION - If significant deviations are noticed, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements, which may be analyte-specific. Check with the project manager about client-specific requirements.

12.10.5 For USACE projects, the LCS would be reanalyzed for the failed analytes only. If the second analysis fails, then the LCS, method blank, and all associated samples of the batch would be re-prepared and reanalyzed for the failed analytes only. If sufficient sample is not available for re-preparation and reanalysis, or if the corrective action is ineffective, the sample results reported within that batch shall be flagged accordingly (*), and a discussion of the impact included within the case narrative.

12.10.6 ACTION – No action is taken on LCS recovery data alone to qualify (e.g. assigning J, I or R flags) associated samples. Professional judgment may be used by the user in evaluating sample data qualification options in conjunction with other QC data for the project.

12.11 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike data are used to evaluate the precision and accuracy of the analytical method on project samples. The MS is evaluated by comparing the recovery for target analytes to the recovery windows established within project documents, or those established by the laboratory. MS data evaluation is more complex than method blank or LCS data evaluation since they measure matrix effects in addition to sample preparation and analysis errors. See the following for potential problems.

12.11.1 The matrix spike analysis is not applicable to samples that contain more than 1000-ppm (mg/L or mg/kg) of the target analyte. According to SW-846 and USACE EM 200-1-3, Appendix I, Section I.10.2.3.1, it is not necessary to perform MS/MSD analyses for the following elements unless superseded by project DQOs:

12.11.1.1 Na, K, Ca, and Mg in aqueous samples.

12.11.1.2 Na, K, Ca, Mg, Fe, Mn, and Al for soil samples.

12.11.2 Since the MS spike levels are intended for low concentration analytes, corrective action is not required for the listed analytes when the MS recoveries are outside the QC limits.

12.11.3 The heterogeneity of soil (NOTE: wet soil samples cannot be homogenized), grab samples, and sequentially collected water samples further complicate the evaluation since matrix-specific bias assumes that the native concentrations in the duplicate analyses are constant. Appropriate homogenization of project samples and subsample collection procedures should be performed to improve representativeness for laboratory analysis. Samples used for matrix duplicate and/or MS duplicates must be homogenized to the extent possible, removing extraneous items (e.g. wood chips, stones, etc. in soil samples) or using samples that appear to be more homogenous (e.g. aqueous samples with little sediment).

12.11.4 If the native concentration of target analytes in the sample chosen for spiking is high relative to the spiking concentration, the differences in the native concentration between the unspiked sample and the spiked samples may not be significant, making the bias measures unrepresentative of the true method and matrix performance. For this reason, if the native concentration is two or more times the spiking level, corrective actions would be based on project DQOS.

12.11.5 In addition to their reluctance to pay for MS and/or MSD analyses, most clients may not know what samples to collect for MS analysis, may not collect sufficient samples, may

not have any knowledge of the native analyte concentrations in the chosen sample, and may not know MS spiking levels that are appropriate either for the sample or their project. For procedures with multiple target analytes, spiking levels may be inappropriate for one or more analytes, if samples that contains target analytes are chosen either in the field or by the laboratory. If no project samples are collected for MS analysis, samples from another project used for MS analysis may not be representative of the project samples. When project samples (e.g. surface soil) are chosen, their MS recovery characteristics may be different from those of other samples (e.g. sub-surface soil) in the same batch.

12.11.6 When MS recoveries fail to meet the MS recovery acceptance criteria, the observed LCS and or LCSD recoveries should be checked to determine the source of difference and to determine if there is a matrix effect or analytical error. Steps may be taken to find the cause of MS failure and corrective actions taken to remedy it. A review of the MSD result, if available, may confirm the matrix effect, if it is the same direction and same order of magnitude. When analytical errors are ruled, sample is homogenous (does not apply to most soil samples), and the native concentration is low, failed MS/MSD recoveries may be used to infer matrix interference.

12.11.7 AML has adopted the SW-846 Method 6010C QC limits for MS/MSD samples. The percent recovery (%R) limits are shown in Tables 6 and 7. Document MS/MSD results in Form-1 equivalents and recoveries/RPD in Form-3 equivalents (see Appendix 12 for details). The %R and RPD should be within QC limits. Check and flag outliers with an asterisk (*).

12.11.8 ACTION - Perform one manual calculation using raw data and extraction sheet information.

12.11.9 ACTION – If MS or MSD recoveries are outside the QC limits, matrix interference should be suspected. Interference checks that consist of serial dilution (SD) and post digestion spike (PDS) analyses. If these tests fail, method of standard addition (MSA) should be performed or analyses performed using an alternate procedure (e.g. ICP/MS) or with instruments with internal standard channels. Sample results based on MSAs may need to be reported for samples that fail MS, MSD, SD and PDS unless re-extraction and reanalyses are performed to address matrix interference problems.

12.11.10 ACTION - Since matrix interferences could lead to serious analytical problems including substantial delays, the supervisor should be consulted when significant outliers are noted. Document QC outliers for %recovery and RPD on the checklist and on client reports (*) and draw attention to the QC outliers on the case narrative. No action may be taken on MS/MSD recovery data alone either to re-extract and/or reanalyze samples. No action may be taken by the laboratory to qualify associated samples. In most cases, no action is taken by the end user to qualify associated data based on MS and/or MSD data. Professional judgment may be used by the user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

12.12 SAMPLE DUPLICATES – Laboratory duplicate analysis can be performed instead of analyzing Matrix Spike (MS) in duplicate. The relative percent difference (RPD) between the duplicate results is used to evaluate the precision of the analytical method on project samples. RPDs are calculated using MS and MSD concentrations (not %R) or sample and sample duplicate concentrations. The MD or MSD is evaluated by comparing the observed

RPDs for target analytes to QC limits (see Tables 6 and 7). These criteria should only be applied to concentrations of target analytes that are above each analyte's MQL. MDs or MSDs that fail to meet the appropriate acceptance criteria may indicate either sample heterogeneity or the presence of potential matrix effect.

12.12.1 The RPDs for sample and MS duplicates ideally should not exceed QC limit.

12.12.2 Check the observed RPD against QC limit. Due to sample heterogeneity, QC outliers are not uncommon. Flag outliers with an asterisk (*).

12.12.3 ACTION – No action is taken on RPD data for soil samples since matrix interferences and inhomogeneity may cause problems. If the LCS is run in duplicate, check the RPD for laboratory control of precision. Document QC outliers for RPD on the checklist and on client reports (*) and draw attention to the QC outliers on the case narrative. Professional judgment may be used by the user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

12.13 SERIAL DILUTION (SD) – Serial dilution is used to evaluate interferences for the analysis as described in Section 11.14. Perform MSA as described in Section 11.16 on one sample from the QC batch, preferably the sample used for MS and MSD analysis. If special problems are noticed for this sample that appears to be uncommon for the remaining samples in the QC batch, use another sample from the batch for this analysis. Samples that contain target analytes at approximately five times the MQL or higher are preferred for this analysis.

12.13.1 The %D between the original result and the 1:5 dilution (1-part sample plus 4-parts diluent) should not excced 10%.

12.13.2 If the original sample results are less than 50 times the MDL (or <10 times the MQL), this test is not valid. If either the diluted sample result or the original sample results are less than 10-times the applicable MDL (or <2 times the MQL), considerable error may result, which may make the results invalid.

12.13.3 Perform SD on another sample, whose results may meet MDL/MQL criteria and which may be representative of the matrix. Flag outliers with an asterisk (*). SD analysis may not be necessary if acceptable post digestion spike (PDS) recoveries have been obtained.

12.13.4 ACTION –Analysis by the method of standard addition (MSA) or analysis using an internal standard may be necessary. Professional judgment may be used by the user in evaluating sample data qualification options in conjunction with other QC data for the project.

12.14 POST DIGESTION SPIKE (PDS) – A PDS analysis is used to evaluate matrix effects in instrumental analysis. It is preferable to have a sample that is either free of or contain relatively low amounts of target analytes. However, in routine practice, it is performed on the sample used for MS, MSD, and SD analyses. If special problems are noticed for this sample that appears to be different from the remaining samples in the QC batch, use another sample from the batch for this analysis. Dilutions of the sample digestates may also be employed to meet spike requirements (See Section 11.14 and 11.15). A PDS analysis may be viewed as the simplest version of the method of standard addition (MSA), which is described in Sections 11.17, 11.18, and 12.15.

12.14.1 The percent recovery for PDS should be within 20% of the true values according to the recent un-promulgated revisions of the SW-846 Method (6010C). The laboratory should transition to the tighter PDS limits.

12.14.2 Check the results for the PDS recoveries to see if the results are within the acceptable range.

12.14.3 ACTION –Perform analysis on a dilution of the sample digestate. Check with the Project Manager if project DQO's can be achieved with analysis performed under dilution.

12.14.4 If acceptable PDS is not attained, perform serial dilution studies, when applicable. Perform MSA as described in Sections 11.17, 11.18 and 12.15 (See also Appendix 7 and SW-846 method 7000 for additional details) or analyze using an internal standard. Consider analysis for the affected analyte using an alternate method (e.g. ICP/MS).

12.15 METHOD OF STANDARD ADDITION (MSA). This procedure is performed in samples that fail SD and PDS tests in addition to failing the percent recovery tests in MS and MSD samples. Perform MSA as described in Section 11.17 and/or 11.8 on one sample from the QC batch, preferably the sample used for MS and MSD analysis. Calculations may be performed either by the instrument or on a PC using Excel spreadsheets. Calculate MSA result as the absolute value of the x-intercept, when the observed result (y-axis) is plotted against spike amounts (x-axis). See Team Leader for details. MSA is often required for critical application (e.g. analysis for waste delisting petitions).

12.15.1 The correlation coefficient for MSA should be equal to or greater than 0.995.

12.15.2 If correlation coefficient is higher, the instrument should be checked for stability and for the accuracy of spike amounts. If the problem cannot be identified, repeat the MSA on a 1:5 dilution of the sample digestate.

12.15.3 Perform MSA on associated samples in the QC batch using the simplified MSA procedure (Section 11.17). Report MSA results with a "D" code for all samples that are associated with the QC sample that fail MS, MSD, SD, and PDS tests.

12.16 SAMPLE RESULTS – Data qualifiers are added by the laboratory during data generation/verification/review. Sample results that contain saturating amounts of any target analyte or analytes that interfere with the target analyte should be re-analyzed under dilution ((1:2, 1:5 or other appropriate dilution). Report sample amounts that are greater than the lowest level for reporting limit (RL, see Table 4) or method detection limit (MDL, see Table 4, 6 and 7 and Appendix 13 for details on MDL studies) as estimated quantities using "J" qualifiers if the levels are less than MQL. (See Tables 6 and 7 for a list of MDLs, RLs, MQLs, etc.). Positive sample results associated with positive MB results should also be qualified with a "B" code. Report sample results in Form-1 equivalents (see Appendix 15 for details). NOTE: CLP packages may require special codes. Check with the Project Manager for project-specific requirements. The following example data qualifiers are suggested for general use.

12.16.1 U - Non-detect when analyte concentration is below MDL or RL as appropriate.

12.16.2 J - Estimated concentration when analyte concentration falls below the MQL.

12.16.3 B - Blank contamination when any associated blanks are above the MDL or RL.

12.16.4 E- Estimated concentration when analyte concentrations exceed the upper limit of the linear range. If the concentration exceeds the linear range of the instrument, dilute and

reanalyze sample digestates for toxic metals. The concentration of base metals (Al, Ca, Mg, Na, Fe, etc.) may be reported without reanalysis under dilution as long as the linear range is not exceeded.

12.16.5D - Data reported are from method of standard addition.

12.16.6I - Invalid results when target and/or non-target interference does not allow a valid result to be reported for a target analyte.

12.16.7R - Rejected due to problems that cannot be resolved by the laboratory within the analytical schedule to report meaningful data for the analyte. This flag can only be used by the Level 3 Reviewer.

13 REFERENCES

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13.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 7000A, Atomic Absorption Methods, July 1992.

13.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 3.2, Sample Preparation Methods, December 1996.

13.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 3.3, Methods for Determination of Inorganic Analytes, December 1996.

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13.7 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3010A, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy, July 1992.

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13.9 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3031, Acid Digestion of Oils for Metals Analysis by Atomic Absorption or ICP Spectrometry, December 1996.

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13.13 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3052, Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices, December 1996.

13.14 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3060A, Alkaline Digestion for Hexavalent Chromium, December 1996.

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13.17 EM 200-1-2, Technical Project Planning (TPP) Process, US Army Corps of Engineers, August 31, 1998.

13.18 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, September 1,1994.

13.19 EM 200-1-6, Chemical Quality Assurance, US Army Corps of Engineers, October 10, 1997.

13.20 ER 1110-1-263, Chemical Data Quality Management for Hazardous, Toxic, Radioactive Waste Remedial Activities, US Army Corps of Engineers, April 30, 1998.

13.21 USEPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, ILM04.0, April 1994.

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13.23 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

13.24 AML Standard Operating Procedures, Q-Water-RC.

13.25 AML Standard Operating Procedures, Q-Subsampling-RC.

13.26 AML Standard Operating Procedures, Q-WasteMgmt-RD.

13.27 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

13.28 AML Standard Operating Procedures, Q-Controlcharts-RC.

Table 1
List of Analytes, Wavelengths, and Method Information

Element	Wavelength (nm)	MDL* (ug/L)	MQL* (ug/L)
Aluminum	308.215	33	100
Antimony	206.833	26	40
Arsenic	193.696	50	70
Barium	455.403	2	5
Boron*	249.678	3.8	15
Beryllium	313.042	0.3	1
Cadmium	226.502	6	6
Calcium	317.933	11	100
Chromium	267.716	10	10
Cobalt	228.616	9	10
Copper	324.754	10	10
Iron	259.940	50	100
Lead	220.353	46	50
Lithium*	670.784	3	10
Magnesium	279.079	24	100
Manganese	257.610	2	5
Mercury*	194.227 (x2)	17	50
Molybdenum	202.030	6	10
Nickel	231.604	8	15
Phosphorus*	213.618	51	200
Potassium	766.491	2420	2500
Selenium	196.026	51	60
Silica*	251.611	17	50
Silver	328.068	13	15
Sodium	588.995	80	200
Strontium*	407.771	0.28	5
Thallium	190.864	21	35
Tin*	189.980 (x2)	17	50
Vanadium	292.402	6	7
Zinc	213.856	4	5

**The Method detection limits (MDLs) and reporting limits (RL) listed above are from EPA method documents. They are for the listed wave lengths only. It is stated in the method that the reporting limits may be higher depending on the type of water and matrix type and they are subject to change.*
** These elements are not monitored at AML using the current equipment. Capability will be available with the addition of a newer equipment (planned for early 2004) with additional channels.*
See other Tables in this SOP for current laboratory MDL, RL, method quantitation limits (MQLs), linear range, etc.

Table 2

Element concentration equivalents arising from interference at the 100 mg/L level Interferent a,b

Element	Wavelength	Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	--	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

-- No interference was observed at the below listed levels

Al - 1000 mg/L
Ca - 1000 mg/L
Cr - 200 mg/L
Cu - 200 mg/L
Fe - 1000 mg/L
Mg -1000 mg/L
Mn - 200 mg/L
Tl - 200 mg/L
V - 200 mg/L

NOTE: Actual interelement corrections (IECs) are dynamic quantities that are subject to change from time to time. Project specific and/or sample-specific IECs may be used by the analyst to improve accuracy of reported data.

Table 3
Concentration of ICV, CCV and MQL Check (CRI) Standards

Analyte	**Concentration (mg/L) of the Standard**		
	ICV	**CCV**	**MQL Check (CRI)**
Aluminum	20.0	10.0	0.40
Antimony	2.0	1.0	0.20
Arsenic	2.0	1.0	0.30
Barium	2.0	1.0	0.010
Beryllium	2.0	1.0	0.010
Cadmium	2.0	1.0	0.020
Calcium	20.0	10.0	0.20
Chromium	2.0	1.0	0.020
Cobalt	2.0	1.0	0.020
Copper	2.0	1.0	0.020
Iron	20.0	10.0	0.20
Lead	2.0	1.0	0.20
Magnesium	20.0	10.0	0.20
Manganese	2.0	1.0	0.010
Molybdenum	2.0	1.0	0.050
Nickel	2.0	1.0	0.050
Potassium	20.0	10.0	5.0
Selenium	2.0	1.0	0.30
Silver	2.0	1.0	0.030
Sodium	20.0	10.0	0.30
Thallium	2.0	1.0	0.30
Vanadium	2.0	1.0	0.020
Zinc	2.0	1.0	0.060

Table 4
Relationship of the Linear Range (LR), Daily High Level Check Standard (HLSTD), Calibration Standard (CAL), CRI Check (MQL), Lowest Level for Reporting (LLR or RL) and Method Detection Limits (MDL) for Aqueous Samples

Analyte	Linear Range (LR) (mg/L)	High Standard (HLSTD) (mg/L)	Calibration Standard (CAL) (mg/L)	CRI Check (MQL) (mg/L)	Lowest Rpt. Level (RL) (mg/L)	Method Det.Lim. (MDL) (mg/L)
Aluminum	1000		100	0.40	0.10	0.049
Antimony	200		10	0.20	0.10	0.034
Arsenic	500		10	0.30	0.15	0.038
Barium	50	20	10	0.010	0.005	0.001
Beryllium	100		10	0.010	0.005	0.001
Cadmium	500		10	0.020	0.005	0.002
Calcium	800		100	0.20	0.10	0.021
Chromium	300	50	10	0.020	0.010	0.002
Cobalt	200		10	0.020	0.010	0.003
Copper	200	50	10	0.020	0.010	0.001
Iron	600		100	0.20	0.10	0.020
Lead	500	100	10	0.20	0.050	0.017
Magnesium	1000		10	0.20	0.10	0.026
Manganese	200	50	10	0.010	0.005	0.001
Molybdenum	250		10	0.050	0.025	0.009
Nickel	750	50	10	0.050	0.015	0.005
Potassium	3000		100	5.0	2.5	0.441
Selenium	250		10	0.30	0.15	0.077
Silver	150		10	0.030	0.010	0.003
Sodium	1000		100	0.30	0.20	0.022
Thallium	250		10	0.30	0.20	0.034
Vanadium	200		10	0.020	0.005	0.002
Zinc	250	50	10	0.060	0.020	0.002

Table 5

SAMPLE HOLDING TIMES, DIGESTED VOLUMES AND COLLECTION VOLUMES FOR METALS DETERMINATION IN AQUEOUS AND SOLID SAMPLES

Measurement	Digested Volume (mL)	Collection Volume (mL)	Holding Time and Preservation
Total	50	600	6 months, HNO_3 to pH <2 Cool to 4°C
Dissolved	50	600	6 months, filter on site; HNO_3 to pH <2 Cool to 4°C
Solid (Total)	1.25-gm	200-g	6 months, Cool to 4°C

Table 6
Aqueous MDLs, MQLs and QC Limits for LCS/LCSD and MS/MSD

Metals Analysis by ICP Atomic Emission Spectroscopy (ICP-AES)

Analytical Management Laboratories

Matrix: Water	Preparation Method:		3010A											
	Analysis Method:		6010B											

	Spike Amount	**Mean B**	S.D.	**MDL A**	Ratio (RD)		**MQL C**	**Ratio (RQ)**		**DoD QSM Control Limits for LCS (%R)**		**SW-846 6010C Control Limits for MS (%R)**		**RPD %**
Element	mg/L	mg/L	mg/L	**mg/L**	RD= B/A	flag	**mg/L**	RQ= C/A	flag	Lower	Upper	Lower	Upper	Limit
Aluminum	0.2	0.221	0.016	**0.049**	4.5		0.400	8.1		80	120	75	125	25
Antimony	0.075	0.078	0.011	**0.034**	2.3		0.200	6.0		80	120	75	125	25
Arsenic	0.15	0.137	0.013	**0.038**	3.6		0.300	7.8		80	120	75	125	25
Barium	0.002	0.002	0.000	**0.000**	5.4	*	0.010	26.9		80	120	75	125	25
Beryllium	0.001	0.000	0.000	**0.000**	2.4		0.010	97.8		80	120	75	125	25
Cadmium	0.01	0.011	0.001	**0.002**	5.1	*	0.020	9.5		80	120	75	125	25
Calcium	0.1	0.103	0.007	**0.021**	4.8		0.200	9.3		80	120	75	125	25
Chromium	0.01	0.011	0.001	**0.002**	5.9	*	0.020	11.1		80	120	75	125	25
Cobalt	0.01	0.011	0.001	**0.003**	3.4		0.020	6.1		80	120	75	125	25
Copper	0.01	0.009	0.000	**0.001**	7.4	*	0.020	15.8		80	120	75	125	25
Iron	0.15	0.156	0.007	**0.020**	7.8	*	0.200	9.9		80	120	75	125	25
Lead	0.05	0.059	0.006	**0.017**	3.5		0.200	11.8		80	120	75	125	25
Magnesium	0.05	0.064	0.009	**0.026**	2.4		0.200	7.6		80	120	75	125	25
Manganese	0.005	0.005	0.000	**0.001**	8.0	*	0.010	15.0		80	120	75	125	25
Molybdenum	0.01	0.012	0.003	**0.009**	1.4		0.050	5.9		80	120	75	125	25
Nickel	0.02	0.017	0.002	**0.005**	3.3		0.050	9.6		80	120	75	125	25
Potassium	5	5.212	0.147	**0.441**	11.8	*	5.000	11.3		80	120	75	125	25
Selenium	0.1	0.116	0.026	**0.077**	1.5		0.300	3.9		80	120	75	125	25
Silver	0.015	0.012	0.001	**0.003**	3.7		0.030	9.1		80	120	75	125	25
Sodium	0.1	0.108	0.007	**0.022**	4.9		0.300	13.5		80	120	75	125	25
Thallium	0.05	0.067	0.011	**0.034**	2.0		0.300	8.9		80	120	75	125	25
Vanadium	0.01	0.010	0.001	**0.002**	5.3	*	0.020	10.5		80	120	75	125	25
Zinc	0.015	0.013	0.001	**0.002**	6.6	*	0.060	30.1		80	120	75	125	25
Mean Ratio (RD or RQ):					4.6			15.0						

Flags (*) are used for mean/MDL ratios (RD) that are <1 or >5 and MQL/MDL ratios (RQ) that are less than 3.

The control and marginal exceedance (ME) limits for LCS are default limits from DoD QSM, Final Revision 2, June 2002.

The QC limits for MS recoveries and RPD are from the USACE EM200-1-3, Appendix I.

RPD limits (25) from USACE EM 200-1-3, Appendix I are greater than the SW-846 6010C limits (20).

ME limits for LCS: Molybdenum, Selenium and Silver (75-120)

Number of ME analytes allowed: One or more analytes with ME limits.

Table 7
Soil MDLs, MQLs and QC Limits for LCS/LCSD and MS/MSD

Metals Analysis by ICP Atomic Emission Spectroscopy (ICP-AES)

Analytical Management Laboratories

Matrix: Soil	Preparation Method:			3050B										
	Analysis Method:			6010B										
	Spike Amount	**Mean B**	S.D.	**MDL A**	Ratio (RD)		**MQL C**	**Ratio (RQ)**		**DoD QSM Control Limits for LCS (%R)**		**SW-846 6010C Control Limits for MS (%R)**		**RPD %**
Element	mg/kg	mg/kg	mg/kg	**mg/kg**	RD= B/A	Flag	**mg/kg**	RQ= C/A	flag	Lower	Upper	Lower	Upper	Limit
Aluminum	10	9.41	0.634	**1.90**	4.9		16.0	8.4		80	120	75	125	25
Antimony	3	2.77	0.558	**1.67**	1.7		8.0	4.8		80	120	75	125	25
Arsenic	4	3.09	0.558	**1.67**	1.8		12.0	7.2		80	120	75	125	25
Barium	0.08	0.070	0.010	**0.029**	2.4		0.400	13.9		80	120	75	125	25
Beryllium	0.04	0.010	0.002	**0.005**	2.2		0.400	85.7		80	120	75	125	25
Cadmium	0.4	0.417	0.046	**0.139**	3.0		0.800	5.8		80	120	75	125	25
Calcium	4	3.88	0.150	**0.450**	8.6		8.00	17.8		80	120	75	125	25
Chromium	0.4	0.437	0.026	**0.079**	5.5		0.800	10.1		80	120	75	125	25
Cobalt	0.4	0.407	0.024	**0.073**	5.6		0.800	10.9		80	120	75	125	25
Copper	0.4	0.369	0.049	**0.148**	2.5		0.800	5.4		80	120	75	125	25
Iron	3	3.42	0.247	**0.741**	4.6		8.00	10.8		80	120	75	125	25
Lead	2	2.14	0.175	**0.526**	4.1		8.00	15.2		80	120	75	125	25
Magnesium	2	2.59	0.202	**0.605**	4.3		8.00	13.2		80	120	75	125	25
Manganese	0.2	0.276	0.018	**0.055**	5.0		0.400	7.3		80	120	75	125	25
Molybdenum	0.4	0.427	0.073	**0.220**	1.9		2.00	9.1		80	120	75	125	25
Nickel	0.8	0.768	0.113	**0.340**	2.3		2.00	5.9		80	120	75	125	25
Potassium	200	200	8.66	**26.0**	7.7		200	7.7		80	120	75	125	25
Selenium	4	4.47	0.843	**2.530**	1.8		12.0	4.7		80	120	75	125	25
Silver	0.6	0.590	0.085	**0.255**	2.3		1.20	4.7		**75**	120	75	125	25
Sodium	10	10.5	1.12	**3.37**	3.1		12.0	3.6		80	120	75	125	25
Thallium	2	2.77	0.404	**1.21**	2.3		12.0	9.9		80	120	75	125	25
Vanadium	0.4	0.361	0.014	**0.041**	8.8		0.800	19.4		80	120	75	125	25
Zinc	1.5	1.30	0.217	**0.652**	2.0		2.40	3.7		80	120	75	125	25
Mean Ratio (RD or RQ):					3.8			12.4						

Flags (*) are used for mean/MDL ratios (RD) that are <1 or >10 and MQL/MDL ratios (RQ) that are less than 3.

The control and marginal exceedance (ME) limits for LCS are default limits from DoD QSM, Final Revision 2, June 2002.

The QC limits for MS recoveries and RPD are from USACE EM200-1-3, Appendix I.

RPD limits (25) from USACE EM 200-1-3, Appendix I are greater than the SW-846 6010C limits (20).

ME limits for Soil LCS: Aluminum, Antimony, Molybdenum, Selenium and Zinc (75-120) and Silver (70-125).

NOTE: The lower control limits for Silver has been lowered from 80 to 75 in LCS (precipitation problems) in DoD QSM.

Number of ME analytes allowed: One or more analytes with ME limits.

Table 8
List of Appendices

Appendix Number	Appendix Topic
1	ICP-AES Calibration Standards Preparation (For Instrument Standardization)
2	Calibration Verification Standards Preparation (ICV, CCV, HLSTD, and MQL Check or CRI)
3	ICP-AES Linear Range Study Summary
4	Interference Check Samples Preparation (ICSA, ICSAB, ICSA2, ICSA3, and ICSAB2)
5	ICP-AES Spike Solution (LCS and MS) Preparation
6	Instrument Run Log Raw Sample and QC Sample Results
7	Method of standard Addition (MSA)
8	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
9	Corrective Action Procedures Summary
10	Client Method Quality Objectives for Method 6010B (AFCEE, USACE, DoD QSM, etc.)
11	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
12	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
13	Method Detection Limit (MDL) Studies Summary
14	Internal Laboratory reports (Excel Spreadsheets) Summary of Sample and QC (MB/LCS/LCSD/MS/MSD/SD/PDS)
15	Sample Reports (Form-1 equivalents)
16	QC Association Forms (Form-4 equivalents) Sample Digestion Logbook Sheets (Soil and Water)

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. I-ICP-MS-Metals-RC

Metals Analysis by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) (EPA 200.8 and SW846 6020A)

October 21, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This Analytical Management Laboratories (AML) standard operating procedure (SOP) is based on Environmental Protection Agency (EPA) SW-846 Method 6020A and EPA contract laboratory program (CLP) Method ILM5.0. This SOP describes a method, which is used to determine trace elements, including metals, in acid solution by inductively coupled plasma mass spectrometry (ICP-MS).

1.2 This SOP is applicable to all elements listed in Table 1 for which EPA has demonstrated acceptability based upon multi-laboratory performance with ICP atomic emission spectrophotometry (ICP-AES) and atomic absorption (AA) based methods.

1.3 The ICP-MS method is applicable to the determination of over 60 elements in sub-µg/L concentrations. This method may be used for the analysis of elements other than those shown on Table 1 if performance is demonstrated.

1.4 This SOP is used routinely for the analysis of elements such As, Sb, Pb, Hg, Se, and Tl, which are commonly determined using GFAA or CVAA technique to achieve lower detection limits.

1.5 Samples consisting of various matrices require digestion prior to analysis with the exception of the analysis for dissolved metals in water samples, which should be filtered and acid-preserved prior to analysis.

1.6 Detection limits, sensitivity, and optimum ranges of metals vary with the matrices and operating conditions. Table 2 shows instrument detection limits (IDL), method detection limits (MDL) and linear range that are normally achievable for aqueous samples using Perkin Elmer ELAN 6100. The manufacturer has provided this information. This information should not be confused with matrix-specific and method-specific data obtained in the laboratory and summarized in Tables 5 and 6.

1.7 An appropriate internal standard is used for each element determined by ICP-MS. Table 3 shows the isotopes monitored and the correction equations that are typically used to determine elemental concentrations using ELAN 6100. The manufacturer has provided this information.

1.8 Analysts performing this method should have either the necessary training and/or prior experience in the operation of ICP/MS and correction of elemental, chemical, and physical interference.

1.9 Routine operational details and detailed maintenance procedures for the ELAN 6100 ICP-MS may be found in the ELAN® Hardware Manual provided by the instrument manufacturer. Such information will not be reproduced in this document.

1.10 Detailed instructions on using ELAN 6100 ICP-MS software may be found in the ELAN 6100 Software Manual. Such information will not be reproduced in this document.

1.11 Detailed information regarding requirements by U.S. EPA SW-846 Method 6020A may be found in Revision 1 (January 1998) of Method 6020, which is included in the EPA website.

1.12 Detailed information regarding requirements by U.S. EPA CLP Method ILM5.0 may be found in the EPA website. Such information will not be reproduced in this document.

2.0 SUMMARY OF METHOD

2.1 Samples are solubilized or digested prior to analysis by using on the methods listed in this SOP, including filtered water samples for dissolved elements.

2.2 Aqueous sample, digestates, leachates, etc. are nebulized into a spray chamber where a stream of argon carries the sample aerosol through a quartz torch and injects it into radio-frequency (RF) inductively coupled plasma (ICP). There the sample is decomposed and desolvated. The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that house a quadrupole mass spectrometer (MS). The ions are sorted according to their mass-to-charge ratio and measured with an electron multiplier detector. Interferences may result from background ions contributed by the plasma gas, reagents, and constituents of the sample matrix. Therefore, appropriate corrections (Table 3) are performed (see also Appendix 2 for details), which allows the analyst to account for these potential interferences. See the following section for additional details.

3.0 INTERFERENCES

3.1 Several types of interferences may cause inaccuracies in the determination of trace elements by ICP-MS. They are described below.

3.2 Abundance Sensitivity. This may result from the wing of a more abundant mass peak contributing to adjacent masses that have considerably lower intensities. This can be minimized from optimizing mass tuning and resolution.

3.3 Isobaric elemental interferences occur when the mass to charge ratio of an isotope of one element is at the same nominal mass to charge ratio of an isotope of another element (e.g., ^{98}Mo and ^{98}Ru). All elements shown in Table 1 with the exception of ^{82}Se (krpton interference) have at least one isotope that is free of isobaric elemental interference. Corrections for isobaric interferences may be made by measuring the intensity due to the interfering element at another isotope and using its natural abundance ratios to correct for its presence at the analytical mass of interest. Most commonly used corrections for isobaric interferences are already present as default interference equations in the ELAN NT software. Care should be taken that the isotope measured for correction purposes does not suffer from overlap with other isotopes that may be present in the sample. A list of

recommended isotopes to be monitored for each element is described in SW-846 Method 6020A (see copy included in Appendix 1 for details).

3.4 Molecular interferences (or isobaric polyatomic ion intereferences) are caused by molecular species formed in the plasma with plasma or matrix ions (examples of common molecular interferences include ArCl, ClO, Nitrogen dimer, oxygen dimer, oxide species, double charged species, etc.) Predictions about the type of molecular interferences may be made using knowledge about the sample matrix. Molecular interferences can often be corrected for in the same manner as isobaric interferences, i.e., measuring the intensity present at another isotope and using isotope ratios to calculate the amount of the interfering species. For example, corrections for interferences of $^{40}Ar^{35}Cl$ on As at mass 75 may be made by measuring the intensity of ArCl present at mass 77 ($^{40}Ar^{37}Cl$) and converting to the apparent intensity of ArCl at mass 75 by using the isotopic ratio of ^{37}Cl to ^{35}Cl. A list of the corrections used is given in the listing of the isotopes monitored in the ELAN NT 6020 method in Table 3 of this SOP. Tables containing isobaric molecular-ion interferences and related information are described in the document, EPA ILM05.0 (see Appendix 2 for copies of four tables from this document).

3.5 Solid phase chelation may be used to eliminate isobaric interferences from both element and molecular sources in samples with low (less than percent) levels of iron and/or aluminum.

3.6 Physical interferences related to sample nebulization, viscosity differences, total solid levels, etc. may result in signal suppression. Although internal standards may compensate for some of these problems, sample dilution (e.g. 1:5) may be necessary.

3.7 Memory interference (or carry-over) from high concentration samples or standards to low concentration samples may result in either false positives or results that are biased higher. The rinse periods between samples should be adjusted to eliminate significant memory interference. Suspected samples should be re-analyzed.

4.0 APPARATUS AND MATERIALS

4.1 Perkin-Elmer ELAN 6100 ICP-MS system.

4.1.1 Includes ELAN 6100 ICP-MS instrument, computer system, ELAN NT software, printer, and Cetac ADX500 Autodiluter autosampler.

4.2 Peristaltic pump tubing:

4.2.1 Black/Black - 0.75 mm i.d. (for sample introduction)
4.2.2 Yellow/Orange - 0.51 mm i.d. (for internal standard introduction)
4.2.3 Purple/White - 2.79 mm i.d. (for drain)
4.2.4 Red/Red - 1.14 mm i.d. (for rinse station)
4.2.5 Blue/Blue - 1.65 mm i.d. (for making connections)
4.2.6 Purple/Purple -2.06 mm i.d. (for making connections)

4.3 T-connector, polypropylene, 1/16 inch i.d. (PE Part # B019-8201; Cole Parmer Part Number: H-06365-77) for hooking up on-line addition of internal standards.

4.4 Calibrated mechanical pipettes:
- 4.4.1 10-100 mL
- 4.4.2 100 - 1000 mL
- 4.4.3 1000 - 5000 mL

4.5 Metal-free plastic pipette tips.

4.6 15-mL plastic AS-91 Autosampler tubes with caps (PE Part # B019-3233).

4.7 50-mL plastic AS-91 Autosampler tubes with caps (PE Part # B019-3234).

4.8 All major maintenance records are logged in the instrument maintenance logbook. Each sample analysis including standards, blanks, field samples, spikes, interference checks are recorded in an instrument run-log book.

4.9 Liquid argon gas supply is of high purity grade.

4.10 Class-A volumetric flasks used to mix and dilute standards to volume.

4.11 Instrument auto sampler tubes made of HDPE plastic, are purchased new and never recycled. Peristaltic pump tubing is replaced at least once a week.

4.12 The plasma torch is cleaned once every 90 days, by turning it upside down in a concentrated Aqua-Regia solution for 2 hours, followed by water rinse.

5.0 **REAGENTS**

5.1 Reagents may contain impurities that may affect the integrity of the analytical results. Due to the high sensitivity of ICP-MS, high-purity reagents, water, and acids must be used whenever possible. All acids used for this method must be of ultra high-purity grade. Nitric acid is preferred for ICP-MS in order to minimize polyatomic interferences. It should be noted that hydrochloric acid is required to maintain stability in solutions containing antimony and silver. When hydrochloric acid is used, corrections for poly-atomic ion interferences must be used. If contamination is found at or above method detection limit, all suspected chemicals are screened individually and disposed of safely.

- 5.1.1 Concentrated Nitric Acid: Ultra-pure from sub-boiling distillation is preferred. Suggested suppliers include: Seastar ™- Sub-boiling distilled grade, Sidney, BC; J. T. Baker - ULTREX® Grade; and Fisher - Optima Grade.
- 5.1.2 Concentrated Hydrochloric Acid: Ultra-pure from sub-boiling distillation is preferred. Suggested suppliers include: Seastar™ - Sub-boiling distilled grade, Sidney, BC; J. T. Baker - ULTREX® Grade; and Fisher - Optima Grade.
- 5.1.3 Reagent water: equivalent to ASTM Type I water (ASTM D 1193). See AML SOP on reagent water for more details.

6.0 **STANDARDS**

The procedure for the preparation of standards has been revised extensively during recent months. This section will be revised again within a short period to reflect actual practice.

6.1 Individual and mixed metal standards for calibration and instrument tuning are purchased from the instrument manufacturer (PE) or other reputable commercial sources.

6.2 Purchase single element stock solutions of the following elements:

6.2.1 Beryllium - PE Part # N930-0172.
6.2.2 Magnesium - PE Part # N930-0179.
6.2.3 Cobalt - PE Part # N930-0113.
6.2.4 Rhodium - PE Part # N930-0144.
6.2.5 Indium - PE Part # N930-0124.
6.2.6 Barium - PE Part # N930-0181.
6.2.7 Cerium - PE Part # N930-0110.
6.2.8 Lead - PE Part # N930-0175.

6.3 Tuning solution Stock: 10-mg/L Be, Mg, Co, Rh, In, Ba, Ce, and Pb.
Prepare by pipetting 500 mL of each 1000 mg/L single element stock solutions (6.6) into a 50-mL autosampler tube. Add 1 mL 1:1 nitric acid and dilute to 50 mL with reagent water.

Note: The suggested solution in Method 6020A does not meet the tuning requirements of the manufacturer for the ELAN 6100. The tuning solution defined in this section of the SOP allows all manufacturer tuning parameters to be met while also meeting the stated requirements of the tuning solution in Section 7.8 of Method 6020A Revision 1 (January 1998).

6.4 Tuning solution: 10-ug/L Be, Mg, Co, Rh, In, Ba, Ce, and Pb.
Prepare by pipetting 1-mL of Tuning Stock solution (6.3) into a 1-Liter flask filled with 200-mL of reagent water, add 10-mL of concentrated nitric acid and dilute to 1-Liter with reagent water and mix well.

6.5 Internal Standard Stock Solution: 10-mg/L ^{6}Li, Sc, Y, In, Rh, Tb, Ho, and Bi; 50-mg/L Ge. Other internal standards may also be used. Higher concentrations are recommended for Sc.

6.5.1 Prepare by pipetting 500 mL of each of the following 1000 mg/L stock solutions into a 50-mL autosampler tube:
6.5.1.1 Rhodium - PE Part # N930-0144.
6.5.1.1 Holmium - PE Part # N930-0123.
6.5.2 Add 5-mL of a stock solution containing 100-mg/L of the following: ^{6}Li, Sc, Y, In, Tb, and Bi (available from VHG Labs, Cat No. LIS-100).
6.5.3 Add 2.5-mL of 1000-mL germanium stock solution (PE Part # N930-0120).
6.5.4 Add 1-mL 1:1 nitric acid and dilute to 50-mL with reagent water.

Note: All solutions including calibration blanks, calibration standards, samples, quality control standards, and quality control samples must be spiked with the same level of the internal standard stock solution. AML procedure for

accomplishing this will be to use on-line addition of the internal standards solution @ 50-ug/L.

6.6 Standard stock solutions. The following combinations are suggested.

6.6.1 Calibration Stock 1 - containing 20-mg/L of Al, As, Sb, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Tl, Th, U, V, and Zn.

6.6.2 Calibration Stock 2 - containing 20-mg/L of Ag and Ba.

6.6.3 Calibration Stock 3 - containing 100-mg/L of Na, Ca, Mg, K, and Fe

6.6.4 Calibration Stock 4 - containing Hg ……..

6.7 ICV (Initial Calibration Verification) stock - the ICV stock (2nd source) will be from the same supplier <u>but from a different Lot#</u> than that used for calibration. The concentrations of the analytes in the ICV are prepared near the mid-point of the calibration range.

6.7.1 ICV Stock 1 - containing 10-mg/L of Al, As, Sb, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Tl, Th, U, V, Zn.

6.7.2 ICV Stock 2 - containing 10-mg/L of Ag and Ba

6.7.3 ICV Stock 3 - containing 100-mg/L of Na, Ca, Mg, K, and Fe

6.7.4 ICV Stock 4 – contains Hg

6.8 Initial Calibration standard: Prepare fresh single or multi-element calibration standards daily as is appropriate for the analysis. The following calibration standard combinations are recommended and have been found to work for a wide variety of samples:

6.8.1 Calibration Blank: A solution containing 1% (v/v) nitric acid in reagent water. Fill a 50-mL autosampler tube with approximately 20-mL of reagent water. Pipette 1-mL of 1:1 (vol/vol) nitric acid into a 50-mL autosampler tube and dilute to 50-mL with reagent water.

6.8.2 Calibration Standard : 10-ug/L of Hg; 100-ug/L of Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Ag, Tl, Th, U, V, and Zn; and 10-mg/L Na, Ca, Mg, K, and Fe. Fill a 50-mL autosampler tube with approximately 20-mL of reagent water. Add 1-mL of 1:1 nitric acid. Pipette 250-µL of Calibration Stock 1, 250-µL of Calibration Stock 2, and 5000-µL of Calibration Stock 3 to the tube, and an appropriate volume of the mercury standard and dilute to 50-mL with reagent water.

6.9 ICV Standard: Prepare fresh daily or as needed for analysis. The following concentrations are suggested when the standards in Section 6.6 are used: 5-ug/L of Hg; 50-ug/L of Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Ag, Tl, Th, U, V, and Zn; and 5-mg/L Na, Ca, Mg, K, and Fe. Fill a 50-mL autosampler tube with approximately 20-mL of reagent water. Add 1-mL of 1:1 nitric acid. Pipette 250-µL of ICV Stock 1, 250-µL of ICV Stock 2, appropriate volume of the Hg standard, and 2500-µL of ICV Stock 3 to the tube and dilute to 50 mL with reagent water.

6.9.1 **Note:** You should also add comparable concentrations of any interfering elements or species, such as chloride, whose intensities may be used in interference corrections to ensure correct equations if the measured intensities are in the analog range of the detector. For example, if the EPA SW-846 3050B digestion is to be measured at a 1:10 dilution, resulting in a hydrochloric acid concentration of 0.5%, the detector cross calibration solution should be made in 0.5% hydrochloric acid.

6.10 CCV Standard: Prepare fresh daily or as needed for analysis as described for ICV. Use the same source as that used for standardizing the instrument.

6.11 Interference Check Solution Stock 1 (ICS Sock 1): This solution contains 1000-mg/L each of Al, Ca, Fe, Mg, Na, P, K, and S; 2000-mg/L of C; 10,000-mg/L Cl; and 20-mg/L each of Mo and Ti.

6.11.1 **Note**: This solution is purchased from a standards vendor providing high quality solutions for ICP-MS.

6.12 Interference Check Solution Stock 2 (ICS Stock 2): This solution contains 10-mg/L each of As, Cd, Cr, Co, Cu, Mn, Ni, Ag, and Zn.

6.12.1 **Note**: This solution is purchased from a standards vendor providing high quality solutions for ICP-MS.

6.13 Interference Check Solution A (ICSA): This solution contains 100-mg/L each of Al, Ca, Fe, Mg, Na, P, K, and S; 200-mg/L of C; 1000-mg/L Cl, and 2-mg/L each of Mo and Ti.

6.13.1 Prepare by diluting the Interference Check Solution Stock 1 tenfold with reagent water. Pipette 5-mL of ICS Stock 1 into a 50-mL autosampler tube and dilute to 50-mL with reagent water.

6.13.2 Prepare this solution fresh daily.

6.14 Interference Check Solution AB (ICSAB): This solution contains 100-mg/L each of Al, Ca, Fe, Mg, Na, P, K, and S; 200-mg/L of C; 1000-mg/L Cl; 2-mg/L each of Mo and Ti; and 0.020-mg/L each of As, Cd, Cr, Co, Cu, Mn, Ni, Ag, and Zn.

6.14.1 Prepare by diluting the Interference Check Solution Stock 1 tenfold with reagent water. Pipette 5-mL of ICS Stock 1 into a 50-mL autosampler tube.

6.14.2 Add 100-µL of Interference Check Solution Stock 2 into the tube, dilute to 50-mL with reagent water.

6.14.3 Prepare this solution fresh daily.

6.15 Laboratory Control Samples are prepared using procedures that are described in the SOP for ICP-AES analysis.

6.16 Matrix Spike Samples are prepared using procedures that are described in the SOP for ICP-AES analysis.

6.17 Blanks: Calibration blank is used during initial standardization, while reagent blank is used to account for acid content variance.

6.17.1 The calibration blank has the same amount of acid as the element calibration standards.

6.17.2 The Method Blank is digested at the same acid level as the unknown samples and spike samples. Method blanks are analyzed as per QA/AC schedule listed in this Standard Operating Procedure or as per regulatory documents governing the batch analysis. The final volume of the unknown samples , spikes and method blanks must be the same.

6.18 Traceability:

6.18.1 All metals standards are to be logged into the Standards and Reagents logbook immediately after they are prepared. The standards are each given a unique reference number based on the date and sequence of preparation (e.g., if two standards were prepared on December 5th 1999, the reference #'s assigned to them would be M991205-01 and M991205-02.

6.18.1.1 All ICP Instrument standards are to be identified in the PE ELAN-6100 software auto-sampler tables by their reference number, book number, and page number.

7.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

7.1 Definitions of terms:

7.1.1 Total Metals: The concentration of metals determined in a sample following digestion.

7.1.2 Suspended Metals: The concentration of metals determined in the portion of a sample that has been filtered through a 2.0 um filter and followed by digestion.

7.1.3 Dissolved Metals: The concentration of metals determined in a sample after the sample is filtered through a 0.45 um filter.

7.1.4 Total recoverable metals: The concentration of metals in an unfiltered sample following treatment with hot dilute mineral acid.

7.2 Sample collection and Preservation: See Table 4.

7.3 One liter plastic bottles with screw caps may be used for collecting aqueous samples in accordance with EPA SW-846 methods 6010B, 6020A, and 7000 series methods. Order pre-cleaned bottles containing the nitric acid preservative directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each water sample to be collected. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. See project specific field sampling plan (FSP) for sample collection and preservation instructions. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.

7.4 One 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples in accordance with EPA SW-846 Method 6010B, 6020A, or 7000 series methods. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each solid sample. Quality control samples such as MS and MSD should be treated as regular environmental samples. Upon receipt and log-in,

these sample containers should be stored in the walk-in cooler. These samples may be shared with samples collected for other analyses such as metals by AA (7000), chlorinated pesticides (8081A), PCBs (8082), other pesticides (8140, etc.), semivolatile organics (8270C) and diesel range organics (DRO, 8015-mod.) or TPH.

7.5 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Analytical data generated outside of the specified holding times must be considered to be minimum values only. Such data may be used to demonstrate that a waste is hazardous where it shows the concentration of a constituent to be above the regulatory threshold but cannot be used to demonstrate that a waste is not hazardous. The holding time for the extraction and/or analysis of aqueous and soil samples (including TCLP leachate preparations) is 6-months from the date of sample collection.

8.0 HEALTH AND SAFETY

8.1 The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory technique and safety practices should be followed at all times.

8.2 Calibration standards and other standards used for quality control contain a number of toxic heavy metals at high concentrations. The toxicity of these complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

8.3 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

8.4 Reagents used such as acids are highly corrosive. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

8.5 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

8.6 Liquid argon represents a potential cryogenic hazard and safe handling procedures should be used when handling liquid argon tanks at all times.

8.7 The ELAN 6100 is fully interlocked to protect the user from dangers such as high voltages, radio frequency generators, and intense ultra-violet light. At no time should the operator attempt to disable these interlocks or operate the ELAN if any safety interlock is known to be disabled or malfunctioning.

8.8 Spilled samples, reagents, and water should be cleaned up from instrument and autosampler surfaces immediately. In the case of acid spills the acid should be neutralized with sodium bicarbonate solution before cleanup.

8.9 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

9.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

9.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

9.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

9.3 Soil samples that have undergone extraction should be free of contaminants. They can be disposed off into sanitary dumpsters. Dispose of sample extracts containing high levels of acid in accordance with applicable AML SOPs.

10.0 PROCEDURE

10.1 Perkin Elmer ELAN 6100: Initiate the plasma and allow a warm-up of at least 30 minutes. The tuning procedures may be carried out during warm-up

10.2 Open the EPA6020 TUNE method and optimize the following parameters using the 10-ug/L tuning solution containing Be, Mg, Co, Rh, In, Ba, Ce, and Pb.

10.2.1 Set RF power to desired level.

10.2.1.1 1000-1100 watts for clean waters.
10.2.1.2 1300-1400 watts for soil and sediment digests.

10.2.2 Optimize the nebulizer argon flow.

10.2.3 Optimize the static lens voltage.

10.2.4 Save the Optimization file.

10.3 Open the EPA6020 LENS CALIB method and perform the autolens calibration using the 10-ug/L tuning solution.

10.3.1 Clear the old calibration

10.3.2 Click on Get Analytes.

10.3.3 Click on Optimize (the procedure takes about 6 minutes).

10.4 Save the Optimization File

10.5 Open the EPA6020 DETECTOR CALIB method and aspirate the dual detector calibration solution (see Section 6.14) containing all elements to be quantitated plus the internal standard elements.

Note: This procedure is only required when detector voltages are changed or a new detector is installed.

10.5.1 Run the detector cross calibration routine.

10.5.1.1 Clear the old calibration.
10.5.1.2 Click on Get Analytes.
10.5.1.3 Click on Optimize.

10.5.2 Save the Optimization File.

10.5.3 Print out the Optimization File (if desired).

10.6 Open the EPA6020 TUNE method

10.6.1 Aspirate the 10-ug/L tuning solution and collect five readings.

10.6.2 Click on the Calibrate button in the Tuning window. Check that the resolution at ^{24}Mg and ^{208}Pb is less than 0.9-amu (full width measured at 10% peak height). Check that the %RSD for the mean mass is less than 5% (advisory). It may be desirable to adjust mass resolution at 5% peak height to be less than 0.75-amu for the instrument to meet required criteria.

10.6.3 Check mass calibration, save the new calibration if the average measured mass is less than 0.1-amu from the true mass.

10.6.4 Print the tuning window using Paintbrush if documentation is required

Note: Using the Clock feature in Windows a data/time stamp can be added to the printout by dragging the clock onto the tuning window.

10.7 Open the EPA6020 DAILY method.

10.7.1 Aspirate the 10-ug/L tuning solution

10.7.2 Click on the Analyze button in manual sample window to acquire

10.7.3 Check that the RSDs for the five replicates for all Be, Mg, Co, In, Rh, and Pb are all less than 5%.

10.7.4 Monitor daily performance measures as recommended by Perkin Elmer for Rh sensitivity, background, % double charged and % oxide levels.

10.7.4.1 Rh > 150,000 cps for 10-ug/L
10.7.4.2 Background at mass 220 < 30 cps
10.7.4.3 % double charged < 3%
10.7.4.4 % oxides < 3%

Note: Oxides and double charged levels can be reduced by slightly decreasing the nebulizer flow rate.

10.8 Open the EPA6020 method.

10.9 Prepare the calibration blank and the calibration standard as described in Section 6.8.

10.10 Make any desired changes to the method and save method under a different name. (The EPA6020.mth method is write protected.)

10.10.1 Load the calibration blank and calibration standard into the autosampler positions specified in the autosampler page of the analytical method.

10.10.2 Load the Quality Control samples defined in the QC checking part of the method into the autosampler according to the positions entered in the QC Autosampler page of the method. (See the QC Manual for more information on setting up Quality Control Protocols.)

10.11 Edit the Sample window for batch analysis to update with new sample information.

10.11.1 Load the applicable method for the sample batch.

Note: If automatic QC checking is used the method must be the same for all samples.

10.11.2 The Calibration action for the first sample for which concentration results are desired must be "analyze blank, standards, and sample"

10.11.3 The calibration action for all other samples is usually "Analyze Sample", unless periodic recalibration is desired.

10.11.4 Enter peri-pump control speeds for all samples

10.11.5 Save Sample file

10.11.6 Re-Open the sample file (this must be done for the batch QC to run properly)

10.11.7 Load the samples into the autosampler positions specified in the Sample file.

10.11.8 Select the samples to be analyzed by highlighting the row number with the mouse

10.11.9 Select "Analyze Batch"

Aspirate the rinse blank for 5-10 minutes before beginning a batch run to avoid carry-over and contamination.

11.0 QUALITY CONTROL

11.1 Quality control (QC) procedures are used to ensure that proper sample preparation technique, extract cleanup procedures, and instrument calibration procedures are performed. The instrument calibration involves the following: mass calibration and resolution check of the ICP-MS instrument; calibrating the instrument for analytes of interest using calibration blanks and calibration standards; initial verification of the calibration with standards and blanks (ICV and ICB); continuing verifications of the calibration during the analysis sequence with standards and blanks (CCV and CCB) for every 10 samples; and final verification of the calibration at the end of sample analysis with standards and blanks (FCV and FCB). It also involves the analysis of method blanks (MB), laboratory control samples (LCS and LCSD), and matrix spikes (MS and MSD). Interference detection procedures such as serial dilution (SD) and post digestion spikes (PDS) are also elements of the QC procedures.

11.2 Establish interference correction factors, linear dynamic range (LDR), instrument detection limits (IDL), method detection limits (MDL), reporting limits (RL), and practical quantitation limits (PQL) for each element of interest.

11.2.1 Instrument detection limits. IDLs are determined in accordance with Section 9.2 of SW-846 Method 6020A and applicable AML SOPs. IDLs are estimated by calculating the average of three IDLs determined on three non-consecutive days (e.g. Monday, Wednesday and Friday). Calculate the standard deviation (S) for seven consecutive measurements of a reagent blank or a standard close to the estimated IDL and multiply the standard deviation by 3.14 to obtain each IDL. Report IDLs in µg/L units. See AML SOP on this subject for details.

11.2.2 Method detection limits (MDL). A standard close in concentration to the estimated IDL or MDL is digested in seven replicates and analyzed on the ICP-MS. Calculate the standard deviation (S) for seven consecutive measurements of the digestate and multiply the standard deviation by 3.14 to obtain MDL (see Appendix 10 for details). The MDL is determined in manner similar to IDL with the exception that it need not be performed in triplicate. Report MDLs in µg/L units for aqueous samples and in mg/kg for solid matrices. See AML SOP on this subject for details.

11.2.3 Method or Practical quantitation limits (MQL). Establish MQLs for each analyte in accordance with the AML SOP on this subject. MQLs are generally defined as the lowest concentration where the measured value is within ±20% of the actual value of the standard. Report MQLs in µg/L units for aqueous samples and in mg/kg for solid matrices.

11.2.4 Establish lowest level for reporting sample data (LLR or RL) for each element in accordance with the AML SOP on this subject. Report RL in µg/L units for aqueous samples and in mg/kg for solid matrices. Typically, LLR is set at one half of the MQL.

11.2.5 Linear dynamic range (LDR). LDRs are primarily detector limited. The upper limit of the linear concentration range is established for each analyte by determining the signal response from a minimum of three different concentrations, one of which is close to the upper limit of the linear range. LDRs are not specifically addressed in either SW-846 Method 6020A or

in Chapter 1 of the SW-846, although they are addressed in EPA 200.8 and ILM05.0. It is defined as the highest concentration where the measured value is within 10% of the actual value of the standard. Perform a new LDR study whenever a new detector is installed, detector voltages are changed, new PA tube is installed in the RF generator or sample introduction mechanism is changed significantly. Document LDR results and summary in method files (see Appendix 12 for details).

11.3 Mass Calibration and Resolution Check - Check on a daily basis that the mass calibration does not differ by more than 0.1-amu and that the mass resolution is less than 0.9-amu at 10% peak height as described in Section 10.6.

11.4 Instrument Sensitivity Check - Analyze tuning standards as described in Section 10.7 to determine day to day performance of the instrument for sensitivity (e.g. internal standard responses), reproducibility (e.g. %RSD for multiple determinations).

11.5 Analyte Calibration - Analyze a calibration blank followed by multiple calibration standards from the lowest concentration level to the highest as described in Section 10.11. Calculate linear regression equations through origin as an option in the ELAN software.

11.6 Method or Practical or method quantitation limits (MQL) - MQLs should be verified by running MQL check samples (CRI). This check should follow ICV analysis. The observed values should be within ±20% of the true values. This is an optional procedure to verify instrument performance and sensitivity.

11.7 Initial calibration verification (ICV). ICVs should be performed after instrument calibration procedures have been completed. The ICV should be within ±10% of the true value. If reanalysis of the ICV is unsatisfactory, recalibration is required. If the problem persists the analysis is halted and instrument maintenance is performed.

11.8 Continuing calibration verification (CCV/FCV). The continuing (CCV) and final calibration verification (FCV) check standard sample is analyzed every 10th sample and at the end of sequence respectively. They should be within ±10% of the true value. Otherwise, data for the previous ten samples may be considered unusable (or estimated depending on the monitoring program) and must be reanalyzed.

11.9 Initial calibration blank (ICB). An ICB is analyzed to verify that no target analytes have been inadvertently introduced into the calibration reagents. ICB analysis must be performed after instrument calibration procedures have been completed. The calibration blanks (e.g. ICB) follow the analysis of calibration verification (e.g. ICV) in the run sequence. Blank criteria vary depending on the analytical program: within ±3*IDL, ±MDL, ±0.5*PQL, etc. The calibration blank results should be within one half of the MQL by default. If reanalysis of the calibration blanks are unsatisfactory, resolution of the problem and recalibration is required. If the problem persists the analysis is halted and the source of contamination determined.

11.10 Continuing calibration blanks (CCB/FCB). The continuing calibration blanks (CCBs) and final calibration blank (FCB) are analyzed every 10th sample (or every 2 hours during the run) and at the end of sequence respectively. The calibration blanks (e.g. CCB) follow the analysis of calibration verification (CCV) in the run sequence. The CCBs criteria are identical to the ICB blank criteria.

11.11 Interference check samples (ICS). Interference check involves the analysis of two solutions: solution A (ICSA), which consists of the interferents and

solution B (ICSAB), which consists of the analytes mixed with the interferents. Interference checks are performed at the beginning and at the end of an analytical run or every 12 hours, whichever is more frequent. The analysis of ICSA/ICSAB should be immediately followed by analysis of a CCV/CCB pair.

11.11.1 QC procedures associated with ICSA and ICSAB samples are not specifically addressed in either SW-846 Method 6020A or in Chapter 1 of the SW-846, although they are addressed in ILM05.0.

11.11.2 In principle, the analytical result for those target analytes not added to the ICSA should meet blank criteria described above. However, obtaining ICSA standards for low level analysis by ICP-MS is very difficult. Most commercially available ICSA standards contain low levels of target analytes (e.g. Pb at 1-ppb, Ni at 3-ppb, etc.).

11.11.3 The observed concentration of interfering and target elements should be within ±20% of the true values. True values for the solutions may be established from the mean of five replicates performed by the laboratory, if it is unavailable from the vendor.

11.12 Laboratory control samples (LCS). The frequency of analysis of LCS analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples. LCS can be run in duplicate to obtain precision (RPD) information. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike.

11.12.1 The laboratory has adopted the Department of Defense Quality Systems Manual (DoD QSM) limits for LCS samples. Although this manual contains limits for ICP-AES analysis, it does not provide limits for ICP-MS. AML has adopted the ICP-AES limits for ICP-MS analyses.

11.12.2 The LCS recoveries should be within the DoD QSM limits (±20% of the true values for most analytes) for spiked blank aqueous and soil samples. The DoD QSM allows marginal exceedance for selected elements that are more difficult to analyze (due to sample digestion issues).

11.12.3 QC limits for solid LCS samples may be obtained from outside sources (e.g. ERA). The QC limits for solid LC samples may be established by the vendors or other regulatory agencies.

11.12.4 When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

11.12.5 Calculate relative percent difference (RPD) for LCS duplicates from the original LCS sample result (S) and the LCS duplicate sample result (D) using the following equation: RPD = (|S-D|)*200/(S+D). The RPD for aqueous and soil duplicates, which includes LCS duplicates should be <20%. AML has adopted the USACE EM 200-1-3, Appendix-I limits for duplicates as default values.

11.13 Matrix spikes - Matrix spike (MS) analysis is always performed in duplicate (MSD). It is preferable to run the MS in duplicate rather than analyzing an unspiked sample in duplicate. The frequency of MS analysis is equivalent to a minimum of 2 (one MS and one MSD) per QC batch of 20 samples or less.

11.13.1 Calculate percent recovery (%R) for MS and MSD analyses from the spiked sample result (SSR), the sample result (SR) and the spike added (SA) as follows: %R = (SSR-SR)*100/SA.

11.13.2 AML has adopted the USACE EM 200-1-3, Appendix-I ICP limits (same as SW-846 limits) for MS recoveries. The MS recoveries for aqueous and soil samples should be within ±25% of the true values (75-125% for %Recovery).

11.13.3 Calculate RPD for MS duplicates as described for the LCS duplicates. The RPD for aqueous and soil MS duplicates should be <25% (USACE EM 200-1-3, Appendix-I criteria for ICP), which is less stringent than SW-846 limits (<20% for RPD).

11.14 Laboratory sample duplicates (LD). The analysis of matrix spike in duplicates (MS and MSD) is equivalent to the analysis of a sample in duplicate. Criteria for RPD are described in the previous section. Additional information on precision will be available through the analysis of LCS in duplicate. Perform sample duplicates if the analytical program requires this analysis. In most cases, the analysis of matrix spike sample in duplicate is sufficient.

11.15 Method blanks (MB) or preparation blanks (PB). The frequency of MB analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples. Method Blank criteria are similar to the criteria for ICBs and CCBs. Report MB results in Form-1 equivalents (see Appendix 11 for details). Report association of project and other QC samples with the MB in Form-4 equivalents or QC association forms (see Appendix 13 for details).

11.16 Serial dilution (SD). This is one of the matrix specific tests for interference. Perform 1:5 dilution of one sample from each analytical batch and compare the results for the two analyses. Samples selected for matrix spike analysis can be selected for routine performance of this test.

11.16.1 If the percent difference (%D) between the initial sample result (I) and the serial dilution result (S) after correction for the dilution is within 10%, the absence of interference is indicated. The %D = (I-S)*100/I.

11.16.2 Perform serial dilution test on sample used for MS/MSD analyses.

11.16.3 Perform serial dilution on another sample, if matrix interference is inferred from failed MS, MSD, and PDS recoveries.

11.16.4 The SD is not valid if the diluted sample results are less than 5 times the MQL and/or the initial (undiluted) value is less than 25 times the MQL (or approximately 10 to 50 times the LLR).

11.17 Post digestion spike (PDS). This is one of the matrix specific tests for interference and it is used in conjunction with the MS recovery data. If MS recoveries are outside the QC limits and/or the dilution test fails or found to be not applicable due to low elemental concentration, post digestion spike analysis will be required. Samples selected for matrix spike analysis should be selected for routine performance of this test. The spike level should be at the same level as LCS and MS samples for low concentration samples. For high concentration samples, appropriate dilutions should be employed and the spike amount should be more than 5 times the original sample concentration.

11.17.1 Calculate percent recovery (%R) from the spiked sample result (SSR), the sample result (SR) and the spike added (SA) as follows: %R = (SSR-SR)*100/SA.

11.17.2 The percent recovery should be within the limits of 75-125% or ±25% of the true value.

11.17.3 Adjust the spike level or the dilution of the sample to attain acceptable PDS spike levels, if the recoveries are not within QC limits.

11.18 Method of Standard Additions (MSA) – This procedure is not applicable to ICP-MS, an internal standard procedure. The MSA can be used when interferences are detected from dilution and recovery tests performed as described above. A known volume of the sample extract (V_x) is spiked with a known volume (V_s) of a standard solution of known concentration (C_s) so that the spiked sample concentration (S_a) is approximately twice the concentration of the unspiked sample (S_b). The unspiked sample is diluted with the same volume (V_s) of reagent water. Both samples are analyzed on GFAA and the unknown sample concentration (C_x) is calculated using the following equation:

$$C_x = \frac{S_b * V_s * C_s}{(S_a - S_b) * V_x}$$

where
V_x = Volume of sample extract
V_s = Volume of standard solution spiked
S_a = Measured concentration of spiked sample
S_b = Measured concentration of the unspiked (diluted) sample
C_s = Concentration of the spiking standard
C_x = Concentration of the unknown sample to be reported

11.18.1 The MSA can be performed using multiple spikes and the C_x calculated graphically using Excel spreadsheets (see Appendix 8 for details). Use at least 3 spiking levels at approximately 50%, 100%, and 150% of the original concentration in addition to the analysis of the diluted sample. The spiked and unspiked samples should contain the same amount of sample extract diluted to the same final volume as shown in Table 2.

11.19 Internal standards. The absolute intensities of all internal standards and their relative responses should be monitored for every analysis. If the absolute intensity of any internal standard in a sample falls below 30% of the intensity of that internal standard obtained during initial calibration, problems related to mass drift, errors in the addition of internal standards or matrix effects should be suspected.

11.19.1 The internal standards are not specifically addressed in detail in either SW-846 Method 6020A or in Chapter 1 of the SW-846, although they are addressed in EPA 200.8 and ILM05.0.

11.19.2 Calculate the percent recovery of internal standard (%R) from the observed sample response (S) as a percentage of the response in the calibration blank (B) as follows: %R = (S*100/B). Use the blank associated with the multi-point initial calibration for the comparison.

11.19.3 The default criteria for internal standard recovery is 30-130%. Other criteria (e.g. 60-125%, 30-120%, etc.) are also used for some programs.

11.19.4 The observed value can decrease with time as the instrument gets degraded from sample analysis. Elevated levels (%R >130%) would indicate the presence of the element used for internal standard in the sample.

11.19.5 If the observed recovery is outside the default window, dilute the sample five-fold (1+4) and reanalyze with the addition of appropriate amounts of internal standards.

11.19.6 The percent recovery should be within the default limits (30-130% or tighter, if possible) for ICB, CCB, and FCB. If they are outside the limits, terminate the analysis, correct the problem, recalibrate and reanalyze associated samples.

11.20 Sample Results and LDR. Any sample that has an element response above the established linearity of the instrument (LDR) is diluted and reanalyzed. Samples containing high levels of interfering elements are carefully screened for false positive or negative reporting.

11.21 Whenever possible, the laboratory should analyze standard reference materials (SRM) and participate in relevant performance evaluation studies.

12.0 DATA CALCULATIONS, VERIFICATION AND VALIDATION

12.1 All calculations necessary to convert raw data (ion counts/second) are performed by the ELAN NT software. The calculated quantities are selected by choosing the desired options in the Report Options screen. The default report option for the ELAN NT 6020 Method is EPA6020 QCReport.rop. If the user desires, this format can be edited and saved under a new name. All calculations necessary to convert raw data (ion counts/second) are performed by the ELAN NT software.

12.2 All calculations performed in the ELAN NT software are based on the ratio of the analyte intensity (cps) to the internal standard intensity (cps). In all calculations where internal standards are used (NOTE: Method 6020A requires the use of internal standards) the ratio of the analyte intensity to internal standard intensity is taken before any other calculation is performed.

12.3 Quality Control Sample results may be checked using the QC Checking features in the ELAN NT software. All values entered in the default ELAN NT Method 6020 should be checked and edited to match the true values used by the laboratory. The modified method can then be saved under a different name. The calculated quantities are selected by choosing the desired options in the Report Options screen. The default report option for the ELAN NT 6020 Method is EPA6020 QCReport.rop. If the user desires, this format can be edited and saved under a new name.

12.4 The following sections will provide guidance for verifying and evaluating data generated by this SOP. It provides procedures to assess if data produced is valid and legally defensible.

12.4.1 Document review on one of the checklists or the case narrative (see Appendix 5 for details) as appropriate.

12.4.2 Clients (e.g. USACE or AFCEE) may have additional requirements that are documented in project-specific QAPP and in method quality objectives (MQOs) documents (see Appendix 14 for details).

12.4.3 MQOs are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may be unattainable in routine use for sample analysis. MQOs are available from a number of agencies: USACE, AFCEE, DoD, etc.

12.4.4 Since USACE MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

12.4.5 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality control requirements. Data generated under these conditions may need to be labeled as screening data. Exceptions and data qualifications necessary should be documented on the checklists and eventually on the case narrative

12.4.6 Provide multiple levels of review. For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist, perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete. For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete. For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director will review the checklists (initial/date as necessary), prepare case narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

12.5 HOLDING TIMES – The validity of analytical data is based on holding times of the samples (see Table 4) from the time of collection to the time of analysis or sample prep.

12.5.1 WATER - The holding times for sample digestion and analysis is 180 days from sampling for all analytes except mercury, which is 28 days. Water samples include TCLP leachates.

12.5.2 SOIL – The holding times for sample digestion and analysis is 180 days from sampling for all analytes except mercury, which is 28 days.

12.5.3 WASTE - The holding times for TCLP leachate preparation is 180 days from sampling for all analytes except mercury, which is 28 days.

12.5.4 Holding times are established by comparing the chain of custody sampling date with the date of the injection.

12.5.5 ACTION – If holding times have been exceeded, contact the client. All positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or invalid (I).

12.6 ICP-MS TUNE - Evaluate the measured mass, peak width at 10% peak height and %RSD for the tuning solution as described in sections 10.6 and 11.3. Tabulate the tune summary in project files (see Appendix 3 for details).

12.6.1 Tune Check – Verify that the observed masses are within 0.1-amu of the true values. Verify that the mass resolution is less than 0.9-amu at 10% peak height.

12.6.2 ACTION- Recalibrate the ICP-MS instrument and reanalyze the tuning solution.

12.6.3 Stability Check-Verify that the %RSDs are less than 5%.

12.6.4 ACTION - Allow additional time for the instrument to become stable and reanalyze tuning solution.

12.7 ICP-MS INITIAL CALIBRATION OR STANDARDIZATION – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the instrument is capable of producing highly accurate data within the calibration range used and acceptable data within the linear dynamic range determined quarterly, semi-annual or other frequency (per monitoring program). Document ICAL by including instrument printouts in project files (see Appendix 4 for details). The laboratory uses a calibration blank and one standard to standardize the instrument. The slope is used to calculate sample amounts. Correlation coefficients are not applicable for this calibration procedure.

12.8 INITIAL AND CONTINUING CALIBRATION (ICV, CCVs and FCV)– ICV and CCVs demonstrate that the calibration standards are of acceptable quality and that the instrument performance has not changed since the generation of ICAL. The continuing and final calibration verification solutions are used to check that the instrument is still operating under the linear range of the initial calibration and has remained stable over the entire analytical sequence.

12.8.1 ICV, CCVs and FCV values should not deviate more than ± 10% of the true value.

12.8.2 ACTION – If QC criteria is not satisfied, correct the problem and reanalyze CCVs and FCV and all associated samples. Associates samples include all samples that precede and follow the affected CCV.

12.8.3 Contact client if corrective action does not resolve problem within the schedule for the project. Positive results for compounds not meeting the criteria may be qualified as estimated (J) by the user (client). Report non-detects without qualifiers and mention these outliners in the case narrative.

12.9 METHOD BLANKS (MB) and CALBRATION BLANKS (ICB, CCBs, and FCB) – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Qualification of sample data to alert blank contamination is performed by the laboratory. Data qualification by client personnel may be based on all available blank results (laboratory MB, calibration blanks, equipment blank, and other field blanks). Criteria for blank is especially confusing due to the multitude of benchmarks (IDL, MDL, PQL) and a lack of coherent EPA policy. Analyst discretion is very important in generating data that are professionally acceptable.

12.9.1 Method blanks and calibration blanks should be free of all target analytes and other interfering non-target analytes. To be acceptable, the default limit for all blanks is one half of the MQL.

12.9.2 The blank results should not exceed one or more of the following criteria for specific programs: ±3*IDL, ±MDL, and ±0.5*PQL. Apply appropriate criteria for the analytical program. Contact the Project Manager for details.

12.9.3 Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples. If levels greater than one half the MQLs are present, inspect and correct the problem. Reanalyze all associated samples. Contact client if other criteria are applicable. Exceptions may include quick turn around projects.

12.9.4 ACTION – All positive results are reported with a “B” code, if contaminants are found in the method blank.

12.9.5 ACTION – If an analyte is detected only in the blank and not in any of the associated samples, then no action is taken.

12.9.6 ACTION – If an analyte is detected in the sample which were also detected in associated method blank at levels greater than one half the MQL, re-digestion and reanalysis may be required if the sample amount is required if the sample amount is less than 10 times the blank level. Other client criteria may be applicable to specific projects.

12.9.7 ACTION – If gross contamination is observed, all affected elements are qualified as invalid (I) and automatic re-digestion and reanalysis should be performed.

12.10 LABORATORY CONTROL STANDARD (LCS) – A LCS analysis is used to evaluate method competence in the absence of matrix effects. The LCS may provide a mechanism to check method efficiency for target compounds without the sample matrix as a factor. Default QC limits are based on limits proposed by the Department of Defense Quality Systems Manual (QSM, Final Version 2, June 2002) for ICP-AES analysis. Other evaluation procedures may include comparison with those determined by project-specific DQOs (See project QAPP, if available) or laboratory statistical control limits. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives.

12.10.1 The LCS and LCSD results are reported in Form-1 equivalents and percent recoveries/RPD are reported in Form-3 equivalents (see Appendix 7 for details). Control and marginal exceedance (ME) limits for LCS percent recoveries and the relative percent difference (RPD) between the LCS and LCSD results are shown in Tables 5 and 6.

12.10.2 ACTION - Initially, the effect of QC failure on the samples should be evaluated. Regardless of this assessment, steps shall be taken to find the source of the problem and correct it. The case narrative shall discuss the corrective action taken and any other information.

12.10.3 When multiple (>10) target analytes are reported (e.g. organic analyses such as SVOCs), the DoD QSM acceptance criteria make allowance for the sporadic marginal failure or marginal exceedances ("ME") for a few target analytes. For elemental analyses, ME is allowed for specific analytes only based on their sample digestion characteristics. This allowance is made only for specific hard-to-analyze elements such as Mo, Se, and Ag in aqueous and soil samples. Allowances for additional elements (Al, Sb, and Zn) in soil samples are included in the QSM.

12.10.4 12.10.4 ACTION - If significant deviations are noticed, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements, which may be analyte-specific. Check with the project manager about client-specific requirements.

12.10.5 12.10.5 For USACE projects, the LCS would be reanalyzed for the failed analytes only. If the second analysis fails, then the LCS, method blank, and all associated samples of the batch would be re-prepared and reanalyzed for the failed analytes only. If sufficient sample is not available for re-preparation and reanalysis, or if the corrective action is ineffective, the sample results reported within that batch shall be flagged accordingly (*), and a discussion of the impact included within the case narrative.

12.10.6 12.10.6 ACTION – No action is taken on LCS recovery data alone to qualify (e.g. assigning J, I or R flags) associated samples. Professional judgment may be used by the user in evaluating sample data qualification options in conjunction with other QC data for the project.

12.11 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike data are used to evaluate the precision and accuracy of the analytical method on project samples.

The MS is evaluated by comparing the recovery for target analytes to the recovery windows established within project documents, or those established by the laboratory. MS data evaluation is more complex than method blank or LCS data evaluation since they measure matrix effects in addition to sample preparation and analysis errors. See the following for potential problems.

12.11.1 The MS and MSD results are reported in Form-1 equivalents and percent recoveries/RPD are reported in Form-3 equivalents (see Appendix 6 for details). QC limits for MS/MSD percent recoveries and the relative percent difference (RPD) between the MS and MSD results are shown in Tables 5 and 6.

12.11.2 The matrix spike analysis is not applicable to samples that contain more than 1000-ppm (mg/L or mg/kg) of the target analyte. According to SW-846 and USACE EM 200-1-3, Appendix I, Section I.10.2.3.1, it is not necessary to perform MS/MSD analyses for the following elements (which are seldom analyzed by ICP-MS) unless superseded by project DQOs:

12.11.3 Na, K, Ca, and Mg in aqueous samples.

12.11.4 Na, K, Ca, Mg, Fe, Mn, and Al for soil samples.

12.11.5 Since the MS spike levels are intended for low concentration analytes, corrective action is not required for the listed analytes when the MS recoveries are outside the QC limits.

12.11.6 The heterogeneity of soil (NOTE: wet soil samples cannot be homogenized), grab samples, and sequentially collected water samples further complicate the evaluation since matrix-specific bias assumes that the native concentrations in the duplicate analyses are constant. Appropriate homogenization of project samples and subsample collection procedures should be performed to improve representativeness for laboratory analysis. Samples used for matrix duplicate and/or MS duplicates must be homogenized to the extent possible, removing extraneous items (e.g. wood chips, stones, etc. in soil samples) or using samples that appear to be more homogenous (e.g. aqueous samples with little sediment).

12.11.7 If the native concentration of target analytes in the sample chosen for spiking is high relative to the spiking concentration, the differences in the native concentration between the unspiked sample and the spiked samples may not be significant, making the bias measures unrepresentative of the true method and matrix performance. For this reason, if the native concentration is two or more times the spiking level, corrective actions would be based on project DQOS.

12.11.8 In addition to their reluctance to pay for MS and/or MSD analyses, most clients may not know what samples to collect for MS analysis, may not collect sufficient samples, may not have any knowledge of the native analyte concentrations in the chosen sample, and may not know MS spiking levels that are appropriate either for the sample or their project. For procedures with multiple target analytes, spiking levels may be

inappropriate for one or more analytes, if samples that contains target analytes are chosen either in the field or by the laboratory. If no project samples are collected for MS analysis, samples from another project used for MS analysis may not be representative of the project samples. When project samples (e.g. surface soil) are chosen, their MS recovery characteristics may be different from those of other samples (e.g. sub-surface soil) in the same batch.

12.11.9 AML has adopted the SW-846 Method 6010C QC limits for MS/MSD samples in the absence of other method guidance (e.g. 6020A).

12.11.10 ACTION – When MS recoveries fail to meet the MS recovery acceptance criteria, the observed LCS and or LCSD recoveries should be checked to determine the source of difference and to determine if there is a matrix effect or analytical error. Steps may be taken to find the cause of MS failure and corrective actions taken to remedy it. A review of the MSD result, if available, may confirm the matrix effect, if it is the same direction and same order of magnitude. When analytical errors are ruled, sample is homogenous (does not apply to most soil samples), and the native concentration is low, failed MS/MSD recoveries may be used to infer matrix interference.

12.11.11 ACTION - Reanalyze the MS/MSD sample/extract at additional dilution (when applicable) to minimize matrix interference. If the matrix effect cannot be resolved, discuss the impact on the data within the case narrative. Re-spiking the sample as outlined below at a higher level (e.g., at two to four times the sample concentration) and reanalysis of the sample may be required for specified projects.

12.12 SAMPLE DUPLICATES – Laboratory duplicate analysis can be performed instead of analyzing Matrix Spike (MS) in duplicate for some client projects. The relative percent difference (RPD) between the duplicate results is used to evaluate the precision of the analytical method on project samples. RPDs are calculated using MS and MSD concentrations (not %R) or sample and sample duplicate concentrations. The MD or MSD is evaluated by comparing the observed RPDs for target analytes to QC limits. These criteria should only be applied to concentrations of target analytes that are above each analyte's MQL. MDs or MSDs that fail to meet the appropriate acceptance criteria may indicate either sample heterogeneity or the presence of potential matrix effect.

12.12.1 The RPDs for sample and MS duplicates ideally should not exceed QC limit.

12.12.2 Check the observed RPD against QC limit. Due to sample heterogeneity, QC outliers are not uncommon. Flag outliers with an asterisk (*).

12.12.3 ACTION – No action is taken on RPD data for soil samples since matrix interferences and inhomogeneity may cause problems. If the LCS is run in duplicate, check the RPD for laboratory control of precision. Document QC outliers for RPD on the checklist and on client reports (*) and draw attention to the QC outliers on the case narrative. Professional judgment

may be used by the user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

12.13 SERIAL DILUTION (SD) – Serial dilution is used to evaluate interferences for the analysis.

12.13.112.13.1 The %D between the original result and the 1:5 dilution (1-part sample plus 4-parts diluent) should not exceed 10%.

12.13.2If the original sample results are less than 50 times the MDL (or <10 times the MQL), this test is not valid. If either the diluted sample result or the original sample results are less than 10-times the applicable MDL (or <2 times the MQL), considerable error may result, which may make the results invalid.

12.13.3Perform SD on another sample, whose results may meet MDL/MQL criteria and which may be representative of the matrix. Flag outliers with an asterisk (*). SD analysis may not be necessary if acceptable post digestion spike (PDS) recoveries have been obtained.

12.13.4ACTION –No action may need to be taken on SD recovery data alone either to reanalyze samples and/or to qualify associated samples for most projects. See PDS results. Professional judgment may be used by the user in evaluating sample data qualification options in conjunction with other QC data for the project.

12.14 POST DIGESTION SPIKE (PDS) – A PDS analysis is used to evaluate method competence in the absence of matrix effects. A single spike that meets requirements can be used. Method of standard addition (MSA), which is described elsewhere (see Appendix 8 for details) can be used for critical application (e.g. delisting petitions) when MS, SD and PDS analyses have failed to demonstrate acceptable accuracy for sample results.

12.14.1The percent recovery for PDS should be within ±25% of the true values.

12.14.2Check the results for the PDS recoveries to see if the results are within the acceptable range.

12.14.3 ACTION –If the recoveries are outside the QC limits, perform PDS recovery study on a diluted sample if the default spike amounts are less than sample amounts. Repeat PDS study on another sample from the same sample set if unknown problems are present on the chosen sample.

12.14.4The Method of standard addition (MSA) as described elsewhere in this document (see Appendix 8 for details) is not applicable to internal standard methods (6020A).

12.14.5ACTION - Contact the client. Professional judgment by the user (client) may be used in evaluating sample data qualification options in conjunction with other QC data for the project if PDS is outside QC limits.

12.15 INTERNAL STANDARDS - Tabulate internal standard intensities (see Appendix 9 for details).

12.15.1 The intensities for internal standards in samples and standards should be within 30-130% of the intensities obtained for the ICV or ICB standard on the same day.

12.15.2 ACTION - If the intensities are lower, evaluate the problem (instrument instability, detector degradation, etc.) and correct it, if possible. If problem cannot be identified, proceed with the analysis.

12.15.3 ACTION - If the intensities are outside the QC limit, evaluate the problem (instrument instability, peristaltic pump malfunction for on-line addition of internal standards, etc.). Correct the problem, if possible.

12.15.4 Perform 1:5 dilution of the sample and perform instrumental reanalysis as described in Section 11.20.

12.15.5 If the problem is not corrected and the instrument appears to have degraded from sample analysis, stop the analysis, restore the instrument to the initial conditions, recalibrate, and repeat sample analysis.

12.16 SAMPLE RESULTS – Report sample amounts that are greater than the lowest limit for reporting (LLR or RL) or method detection limit (MDL) using "J" qualifiers if the levels are less than MQL. See Tables 5 and 6 for a list of MDLs, MQLS, etc. Positive sample results associated with positive MB results should also be qualified with a "B" code. If the concentration exceeds the linear dynamic range (LDR) of the instrument, dilute and reanalyze sample digestates. Report sample data in Form-1 equivalents (see Appendix 11 for details).

13.0 **REFERENCES**

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13.9 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3015, Microwave Assisted Acid Digestion of Aqueous Samples and Extracts, September 1994.

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13.20 "Methods for the Determination of Metals in Environmental Samples - Supplement 1", EPA-600/R-94-111, May 1994, Available at NTIS, PB 94-184942, Method 200.8.

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13.22 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

13.23 AML Standard Operating Procedures, Q-Water-RC.
13.24 AML Standard Operating Procedures, Q-Subsampling-RC.
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13.27 AML Standard Operating Procedures, Q-Controlcharts-RC.

Table 1
List of Applicable Elements

Number	Element	Symbol	Chemical Abstracts Service Registry Number (CASRN)
1	Aluminum	Al	7429-90-5
2	Antimony	Sb	7440-36-0
3	Arsenic	As	7440-38-2
4	Barium	Ba	7440-39-3
5	Beryllium	Be	7440-41-7
6	Cadmium	Cd	7440-43-9
7	Calcium	Ca	7440-70-2
8	Chromium	Cr	7440-47-3
9	Cobalt	Co	7440-48-4
10	Copper	Cu	7440-50-8
11	Iron	Fe	7439-89-6
12	Lead	Pb	7439-92-1
13	Magnesium	Mg	7439-95-4
14	Manganese	Mn	7439-96-5
15	Mercury	Hg	7439-97-6
16	Nickel	Ni	7440-02-0
17	Potassium	K	7440-09-7
18	Selenium	Se	7782-49-2
19	Silver	Ag	7440-22-4
20	Sodium	Na	7440-23-5
21	Thallium	Tl	7440-28-0
22	Vanadium	V	7440-62-2
23	Zinc	Zn	7440-66-6

Table 2
Typical ELAN 6100 IDLs, MDLs, and Linear Ranges for Method 6020A

Analyte	Mass	IDL (ug/L)	MDL (ug/L)	Linear Range (mg/L)
Al	27	0.04	0.12	10
Sb	123	0.003	0.03	10
As	75	0.06	0.2	5
Ba	135	0.01	0.01	10
Be	9	0.02	0.02	10
Cd	114	0.002	0.004	5
Cr	52	0.1	0.5	10
Co	59	0.002	0.002	10
Cu	65	0.005	0.02	10
Pb	208	0.001	0.009	10
Mn	55	0.004	0.009	10
Hg				
Ni	60	0.007	0.04	10
Ag	107	0.03	0.03	5
Tl	205	0.0003	0.0003	10
Zn	66	0.015	0.03	5
Se*	82	0.09	0.09	5
Mo*	98	0.004	0.004	5
Th*	232	0.002	0.002	10
U*	238	0.0009	0.0009	10
V*	51	0.03	1.2	10
Na*	23	0.6	4	100
Ca*	44	15	20	200**
Mg*	24	0.02	0.04	100
K*	39	9	9	100
Fe*	54	4	4	200**

*For information only.
**Highest level standard ran for linearity test.

Table 3
Table of Isotopes Monitored and Equations Used

Analyte	Symbol	Isotopes Monitored	Correction Equations
Aluminum	Al	27	
Antimony	Sb	121,**123**	Sb 123 = Sb 123 - 0.127189 * Te 125
Arsenic	As	75	As 75 = As 75 - 3.127 * [ArCl 77 - (0.815*Se 82)]
Barium	Ba	**135**,137	
Beryllium	Be	9	
Cadmium	Cd	106,108,**111,114**	Cd 111 = Cd 111 - 1.073 * [MoO 108 -(0.712* Pd 106)] Cd 114 = Cd 114 - 0.026826 * Sn 118
Chromium	Cr	**52,53**	
Cobalt	Co	59	
Copper	Cu	**63,65**	
Lead	Pb	**206,207,208**	Pb 208 = Pb 208 + 1* Pb 206 + 1* Pb 207
Manganese	Mn	55	
Nickel	Ni	**60**,62	
Silver	Ag	**107,109**	
Thallium	Tl	203,**205**	
Zinc	Zn	**66,67,68**	
Internal Standards			
Lithium	Li	6	
Scandium	Sc	45	
Yttrium	Y	89	
Rhodium	Rh	103	
Indium	In	115	
Terbium	Tb	159	
Bismuth	Bi	209	
Germanium	Ge	72	
(Information	**Only)**		
Molybdenum	Mo	95,97,98	Mo 98= Mo 98 - 0.110588 * Ru 101
Selenium	Se	**77,82**	Se 82 = Se 82 - 1.008696 * Kr 83
Thorium	Th	232	
Uranium	U	238	
Vanadium	V	51	V 51 = V 51 - 3.127*[ClO 53 - (0.113*Cr 52)]
Calcium	Ca	44	
Magnesium	Mg	24	
Sodium	Na	23	
Potassium	K	39	
Iron	Fe	54	Fe 54 = Fe 54 - 0.028226 * Cr 52
Mercury	Hg	202, **200**, 199, 201	

Table 4

SAMPLE HOLDING TIMES, DIGESTED VOLUMES AND COLLECTION VOLUMES FOR METALS DETERMINATIONS IN AQUEOUS AND SOLID SAMPLES

Measurement	Digested Volume** Or weight (mL/g)	Collection Volume or weight (mL/g)	Holding Times* and Preservation
Total	50	600	6 months, HNO3 to pH <2 cool to 4°C
Dissolved***	50	600	6 months, filter on site; HNO3 to pH; cool to 4°C
Suspended	50	600	6 months, filter on site cool to 4°C
Solid (Total)	1.25-g	200-g	6 months, cool to 4°C

*Holding time for mercury (Hg) is 28-days from sample collection.
**The sample amounts are 10-mL and 0.6-g respectively for mercury.
***Sample digestion is not necessary for this analysis.

Table 5
Aqueous MDLs, MQLs and QC Limits for LCS/LCSD and MS/MSD

Metals Analysis by ICP Mass Spectrometry (ICP-MS)														
Analytical Management Laboratories														
Matrix: Water	Preparation Method			3020A										
	Analysis Method			6020A										
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**		**LCS Limits**		**MS Limits**		**RPD**
Element	Amount	ug/L	ug/L	**ug/L**	RD=B/A	flag	**ug/L**	RQ=C/A	flag	Lower	Upper	Lower	Upper	Limit
	ug/L	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%
Aluminum	20	32.3	3.250	**10.2**	3.2		100	9.8		80	120	75	125	25
Antimony	0.10	0.109	0.003	**0.010**	10.4	*	5.0	477.0		80	120	75	125	25
Arsenic	*1.0*	*0.489*	*0.076*	**0.240**	2.0		3.0	12.5		80	120	75	125	25
Barium	0.3	0.471	0.016	**0.049**	9.6	*	1.0	20.3		80	120	75	125	25
Beryllium	0.5	0.613	0.050	**0.157**	3.9		1.0	6.4		80	120	75	125	25
Cadmium	0.2	0.256	0.024	**0.075**	3.4		1.0	13.2		80	120	75	125	25
Calcium	100	133	6.486	**20.4**	6.5	*	100	4.9		80	120	75	125	25
Chromium	1	1.479	0.167	**0.523**	2.8		2.0	3.8		80	120	75	125	25
Cobalt	0.1	0.062	0.007	**0.023**	2.7		1.0	42.9		80	120	75	125	25
Copper	*3.0*	*4.444*	*0.411*	**1.292**	3.4		5.0	3.9		80	120	75	125	25
Iron	20	36.3	6.627	**20.8**	1.7		100	4.8		80	120	75	125	25
Lead	0.2	0.267	0.056	**0.177**	1.5		1.0	5.7		80	120	75	125	25
Magnesium	10	16.7	0.897	**2.817**	5.9	*	100	35.5		80	120	75	125	25
Manganese	0.5	0.761	0.025	**0.080**	9.5	*	1.0	12.5		80	120	75	125	25
Mercury	0.2	0.310	0.014	**0.045**	6.9	*	0.50	11.2		80	120	75	125	25
Nickel	0.1	0.212	0.027	**0.090**	2.3		2.0	22.1		80	120	75	125	25
Potassium	30	48.7	6.124	**19.2**	2.5		100	5.2		80	120	75	125	25
Selenium	1	1.276	0.117	**0.368**	3.5		2.0	5.4		80	120	75	125	25
Silver	0.1	0.120	0.008	**0.027**	4.5		5.0	188.7		80	120	75	125	25
Sodium	20	68.0	11.9	**37.4**	1.8		200	5.3		80	120	75	125	25
Thallium	0.2	0.255	0.010	**0.030**	8.5	*	1.0	33.4		80	120	75	125	25
Vanadium	2	2.894	0.214	**0.673**	4.3		5.0	7.4		80	120	75	125	25
Zinc	2	2.080	0.183	**0.615**	3.4		20	32.5		80	120	75	125	25
Mean Ratio (RD or RQ):					4.5			41.9						
Flags (*) are used for mean/MDL ratios (RD) that are <1 or >5 and MQL/MDL ratios (RQ) that are less than 3.														
The control and marginal exceedance (ME) limits for LCS are default limits from DoD QSM, Final Revision 2, June 2002.														
The QC limits for MS recoveries and RPD are from the USACE EM200-1-3, Appendix I.														
RPD limits (25) from USACE EM 200-1-3, Appendix I are greater than the SW-846 6010C limits (20).														
ME limits for LCS: Selenium and Silver (75-120)														
Number of ME analytes allowed: One or more analytes with ME limits.														

Table 6
Soil/Sediment MDLs, MQLs and QC Limits for LCS/LCSD and MS/MSD

Metals Analysis by ICP Mass Spectrometry (ICP-MS)														
Analytical Management Laboratories														
Matrix: Soil	Preparation Method			3050B										
	Analysis Method			6020A										
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**		**LCS Limits**		**MS Limits**		**RPD**
Element	Amount	mg/kg	mg/kg	**mg/kg**	RD=B/A	flag	**mg/kg**	RQ=C/A	flag	Lower	Upper	Lower	Upper	Limit
	mg/kg	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%
Aluminum	8	20.10	3.340	**10.5**	1.9		40	3.8		80	120	75	125	25
Antimony	0.04	0.07	0.011	**0.036**	2.0		2.0	56.1		80	120	75	125	25
Arsenic	0.8	0.46	0.116	**0.36**	1.3		1.20	3.3		80	120	75	125	25
Barium	0.12	0.182	0.012	**0.037**	4.9		0.40	10.7		80	120	75	125	25
Beryllium	0.2	0.243	0.020	**0.061**	4.0		0.40	6.5		80	120	75	125	25
Cadmium	0.08	0.154	0.018	**0.058**	2.6		0.40	6.9		80	120	75	125	25
Calcium	40	55.12	1.715	**5.39**	10.2	*	40	7.4		80	120	75	125	25
Chromium	0.4	1.597	0.079	**0.250**	6.4		0.80	3.2		80	120	75	125	25
Cobalt	0.04	0.032	0.002	**0.007**	4.8		0.40	61.0		80	120	75	125	25
Copper	0.16	0.285	0.012	**0.041**	7.0		2.0	49.1		80	120	75	125	25
Iron	8	40.21	0.774	**2.43**	16.5	*	40.0	16.5		80	120	75	125	25
Lead	0.08	0.11	0.017	**0.055**	2.1		0.40	7.3		80	120	75	125	25
Magnesium	4	6.26	0.214	**0.67**	9.3		40	59.6		80	120	75	125	25
Manganese	0.2	0.298	0.012	**0.037**	8.1		0.40	10.9		80	120	75	125	25
Mercury	0.08	0.110	0.004	**0.012**	9.4		0.20	17.2		80	120	75	125	25
Nickel	0.04	0.069	0.006	**0.021**	3.3		0.80	38.2		80	120	75	125	25
Potassium	12	18	0.80	**2.5**	7.1		40	15.9		80	120	75	125	25
Selenium	0.4	0.45	0.072	**0.23**	2.0		0.80	3.5		80	120	75	125	25
Silver	0.04	0.099	0.006	**0.018**	5.7		2.0	114.2		**75**	120	75	125	25
Sodium	4	41.3	2.54	**7.96**	5.2		80	10.0		80	120	75	125	25
Thallium	0.08	0.09	0.004	**0.012**	7.7		0.40	33.9		80	120	75	125	25
Vanadium	0.8	0.479	0.199	**0.63**	0.8	*	2.0	3.2		80	120	75	125	25
Zinc	0.8	0.16	0.212	**0.71**	0.2	*	8.0	11.2		80	120	75	125	25
Mean Ratio (RD or RQ):					5.3			23.9						
Flags (*) are used for mean/MDL ratios (RD) that are <1 or >10 and MQL/MDL ratios (RQ) that are less than 3.														
The control and marginal exceedance (ME) limits for LCS are default limits from DoD QSM, Final Revision 2, June 2002.														
The QC limits for MS recoveries and RPD are from USACE EM200-1-3, Appendix I.														
RPD limits (25) from USACE EM 200-1-3, Appendix I are greater than the SW-846 6010C limits (20).														
ME limits for Soil LCS: Aluminum, Antimony, Selenium and Zinc (75-120) and Silver (70-125).														
NOTE: The lower control limits for Silver has been lowered from 80 to 75 in LCS (precipitation problems) in DoD QSM.														
Number of ME analytes allowed: One or more analytes with ME limits.														

Table 7
List of Appendices

Appendix Number	Appendix Topic
1	Reference Material from SW-846 Method 6020A Recommended Isotopes for Analyte Elements
2	Reference Materials (Attached Tables) from EPA ILM05.0 Isobaric molecular ion interferences for ICP-MS Analysis Recommended mass choices for elements Recommended elemental expressions for isobaric interferences Recommended internal standards
3	Stanadards Preparation for ICP-MS Analysis ICP-MS Tuning
4	Instrument Printouts ICP-MS Instrument Calibration ICP-MS Calibration Verification
5	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
6	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
7	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
8	Method of Standard Additions (MSA) Reference Material from EPA SW-846
9	ICP-MS Internal Standards Summary
10	Method Detection Limit (MDL) Studies Summary
11	Sample Reports (Form-1 equivalents)
12	ICP-MS Linear Range Studies Summary
13	QC Association Forms (Form-4 equivalents) Sample Digestion Logbook Sheets (Soil and Water)
14	Client Method Quality Objectives for Method 6020A (AFCEE, USACE, DoD QSM, etc.)

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. I-Mercury-CVAA-RD

MERCURY IN SOIL AND WATER BY COLD VAPOR ATOMIC ABSORPTION (CVAA) TECHNIQUE (EPA 245.1, 245.2, 245.5, SW-846 7470A, and 7471B)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) is based on Environmental Protection Agency (EPA) 245.1, 245.2, 245.5, SW-846 Methods 7470A, 7471A/7471B, and EPA Contract Laboratory Program (CLP) methods ILM04.0, ILM05.0, etc. This SOP is a cold-vapor atomic absorption procedure for determining the concentration of mercury in wastewater, drinking water, mobility-procedure extracts, aqueous wastes, ground water, soil, sediment, and waste samples. All samples must be subjected to an appropriate dissolution step prior to analysis.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, the solid and liquid samples must be prepared in accordance with procedures discussed in this document.

2.2 This SOP is based on EPA methods that employ cold-vapor atomic absorption techniques are based on the absorption of radiation at 253.7-nm by mercury vapor. The mercury is reduced to the elemental state and aerated from the solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height or peak area) is measured as a function of mercury concentration.

2.3 The typical quantitation limit (MQL) for this method is 0.0002 mg/L (0.2-µg/L) in aqueous samples and 0.05 mg/kg in soil samples.

3.0 INTERFERENCES

3.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

3.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

3.3 Seawaters, brines, and industrial effluents high in chlorides require additional permanganate because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater by using this technique.

3.4 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

4.0 APPARATUS

4.1 Mercury Analyzer: Two mercury analyzer instruments are used:

4.1.1 Leeman Labs Hydra AA Automated Mercury Analyzer. This instrument is equipped with a Model PS Autosampler and the WinHg software for data processing. The instrument is rated for detection limits as low as 6.5-ppt in clean aqueous samples.

4.1.2 Spectro Products Analyzer - Model HG-4 with a 22 cm flow cell, Iron Reference (lamp B), and Mercury @ 254 nm Analysis (lamp A). This instrument is equipped with a Technicon autoanalyzer, an Alpkem auto sampler, PC-based chromatography data systems for height or area measurements of the absorbance, and a 10-cm long absorption cell with quartz end windows. The cell can be positioned and adjusted vertically and horizontally to give the maximum transmittance. This instrument is also equipped with the following accessories:

4.1.2.1 Air pump: Any peristaltic pump capable of delivering 1-liter air/min may be used. A Masterflex pump with electronic speed control has been found to be satisfactory.

4.1.2.2 Flowmeter: Capable of measuring airflow of 1 liter/min.

4.1.2.3 Aeration tubing: A straight glass frit with a coarse porosity. Tygon tubing is used for passage of the mercury vapor from the sample bottle to the absorption cell and return.

4.1.2.4 Drying tube: 6-in. x 3/4-in.-diameter tube containing either a resin or magnesium perchlorate to prevent condensation of moisture inside the cell.

4.2 The cold-vapor generator is assembled according to the instrument manufacturer instructions for both instruments.

4.3 The newer Leeman Lab instrument is the primary analysis instrument. The Spectro Products Analyzer serves as a back up. Procedures based on ICP-MS analysis is also used extensively for mercury analysis at AML due to additional qualitative confirmation of the presence of mercury due to quantitative results for Hg from four m/z channels (199, 200, 201, and 202) by the ICP-MS method.

5.0 MATERIALS

5.1 Digestion Tubes: 50-ml Environmental Express vials for soil samples.
5.2 Test Tubes: 16x100 mm test tubes with lids for aqueous samples.
5.3 Test Tube Racks: Polypropylene racks adequate to hold test tubes.
5.4 Water Bath or Oven: Waterbath (capable of heating tubes to 95°C).
5.5 Pipettes

6.0 REAGENTS

6.1 Sulfuric Acid, conc.: Reagent Grade, Baker Instra-analyzed or equivalent.

6.2 Nitric Acid, conc.: Reagent Grade, Baker Instra-analyzed or equivalent.
6.3 Hydrochloric Acid, conc.: Reagent Grade, Baker Instra-analyzed or equivalent.
6.4 Stannous Chloride: 4% $SnCL_2$ and 5% HCL. Add 16-g of $SnCL_2$ to a solution containing 375-mL deionized water and 20-mL concentrated HCl. Mix for several hours to allow all of the material to enter the solution. Store it in a PVC container. Use a magnetic stirrer to constantly mix the solution when it is being used. Keep the container closed when it is not in use. If the above procedures are followed, the solution is good for at least a week. When the cold vapor is generated manually, mix 5-mL of this reagent with the sample digestates. An equivalent quantity of this reagent is mixed by the instruments to generate cold mercury vapor from digestates.
6.5 12% w/v Sodium Chloride-Hydroxylamine Hydrochloride Solution: Dissolve 120-gm NaCl and 120-gm NH_2OH HCl to 1 L using deionized water. Store in a PVC container and mix well before using.
6.6 3% w/v Sodium Chloride-Hydroxylamine Hydrochloride Solution*:* Add 250-mL 12% NaCl-NH_2OH HCl to 750-mL deionized water. Put in a PVC container and mix well before using.
6.7 Potassium Permanganate Solution: 5% Solution, s/v. Dissolve 50-gm $KMnO_4$ in 1-L of deionized water. Store in a PVC container.
6.8 Acid Wash Reagent: 3% Each in HCl and HNO_3. To 940-mL of deionized water, add 30-mL concentrated HCl and 20-mL concentrated HNO_3. Mix well and store in a PVC container.
6.9 Potassium Persulfate Solution: 5% solutions, w/v. Dissolve 50-gm $K_2S_2O_8$ in 1-L deionized water. Store in a PVC container.
6.10 Stock Mercury Standard Solution: A stock solution is ordered through Ultra Scientific (cat # ICP-080, 1000-µg/mL) or equivalent as certified solutions. A 1000 µg/mL Hg standard is used for the stock solution. OR: Dissolve 0.1254-g of $HgCl_2$ in 60-mL deionized water in a 100-mL volumetric flash. Add 10-mL concentrated HNO_3 and 10-mL concentrated HCl, mix well, and dilute to 100.0-mL with deionized water. 1 mL = 1 mg Hg. Stock solutions expire on the day specified by the manufacturer. Stock solutions are stable up to two years.
6.11 Intermediate Mercury Calibration Standard Solution: Prepare intermediate mercury standard solution at a concentration of 100-µg/mL using a two-step dilution process. Dilute 1.0-mL of the Stock Solution (Section 6.10) to 100-mL with nitric acid (3-mL) and deionized water so that the nitric acid concentration is 3%. Dilute 1.0-mL of the resulting solution (10-µg/mL) to 100-mL with nitric acid (3-mL) and deionized water so that the nitric acid concentration is 3%. This final intermediate solution (0.1-µg/mL or 100-µg/L) is used to prepare the daily working standards (initial calibration and calibration verification). The final intermediate standard is replaced one year after preparation or sooner if it is used up.
6.12 Working Mercury Standards-aqueous samples: Make dilutions of the intermediate mercury calibration standard solution (Section 6.11) to obtain working standards and QC samples of desired strength as described below:
6.12.1 Prepare 10-mL of aqueous initial calibration (ICAL) solutions by diluting 0.000-mL (calibration blank), 0.020-mL, 0.050-mL, 0.100-mL, 0.200-mL, 0.500-mL and 2.00-mL of the 100-µg/L intermediate standard to 10-mL

with appropriate volumes of deionized water. The lowest calibration standard (0.2-µg/L) is mandatory. The concentration of the higher standards may be adjusted by the analyst so that six levels of standards are prepared and analyzed.

6.12.2 Prepare aqueous calibration verification solutions (CCVs) by diluting 0.2-mL of the appropriate 100-µg/L standard with 9.8-mL deionized water.

6.12.3 Prepare aqueous method blank (MB) and continuing calibration blank (CCB) samples in the same manner as the calibration blank.

6.12.4 Prepare these solutions fresh daily.

6.13 Working Mercury Standards-solid and waste samples: Make dilutions of the intermediate mercury calibration standard solution (Section 6.11) to obtain working standards and QC samples of desired strength as described below:

6.13.1 Prepare 5-mL of soil initial calibration (ICAL) solutions by diluting 0.000-mL (calibration blank), 0.10-mL, 0.25-mL, 0.50-mL, 1.00-mL, 2.50-mL and 5.00-mL of the 100-µg/L intermediate standard to 5-mL with appropriate volumes of deionized water. The lowest calibration standard is mandatory. The highest calibration standard does not require any dilution. The concentration of the higher standards may be adjusted by the analyst so that six levels of standards are prepared and analyzed.

6.13.2 Prepare soil calibration verification solutions (CCVs) by diluting 1.0-mL of the 100-µg/L intermediate standard with 4.0-mL deionized water.

6.13.3 Prepare soil method blank (MB) and continuing calibration blank (CCB) samples in the same manner as the calibration blank.

6.13.4 Prepare these solutions fresh daily.

6.14 Laboratory Control Sample (LCS) Standards: The 100-µg/L intermediate standard (Section 6.11) solution is also used to prepare the aqueous and soil LCS samples as described below:

6.14.1 Water LCS – The aqueous LCS (or LCSD) is prepared by diluting 0.2-mL with 9.8-mL deionized water.

6.14.2 Soil LCS - The soil LCS (or LCSD) is prepared by diluting 1.0-mL with 4-mL deionized water.

6.15 Matrix Spike (MS) Standards: The matrix spike (MS) and MS duplicate (MSD) samples are also prepared using the100-µg/L intermediate standard (Section 6.11) solution as described below:

6.15.1 Use 9.8-mL of the sample instead of the deionized water and prepare aqueous MS samples in a manner similar to that of LCS.

6.15.2 Fortify 0.6-g of the soil sample with 1.0-mL of the standard and 4-mL of the deionized water to prepare soil MS and MSD samples. Use sample weights for MS and MSD that are within 1 to 2% of the sample weights used for the unspiked sample.

6.16 Initial Calibration Verification Samples – These are prepared from a second source stock LCS solution that is ordered through Solutions Plus (cat # 5903, 1000-µg/mL), or an equivalent second source. Dilute two successive 1:100 (1-mL to 100-mL) dilutions the Stock Solution with nitric acid and deionized water so that the intermediate second source standard solution has a final mercury concentration of

100-µg/L as described in section 6.11.

6.16.1 Aqueous ICV Standard-Prepare the aqueous ICV standard by diluting 0.2-mL of the100-µg/L second source standard with 9.8-mL deionized water as described for the preparation of aqueous CCVs in Section 6.12.2.

6.16.2 Soil ICV Standard- Prepare the soil ICV standard by diluting 1.0-mL of the100-µg/L second source standard with 4.0-mL deionized water as described for the preparation of soil CCVs in Section 6.13.2.

6.17 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

7.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

7.1 Definitions of terms:

7.1.1 Total Metals: The concentration of metals determined in a sample following digestion.

7.1.2 Dissolved Metals: The concentration of metals determined in an aqueous sample after the sample is filtered through a 0.45 um filter.

7.2 Sample collection and Preservation: See Table 1.

7.3 One liter high density polyethylene (HDPE) plastic bottles with screw caps may be used for collecting aqueous samples in accordance with EPA SW-846 methods 6010B and 7000 series methods. Order pre-cleaned bottles containing the nitric acid preservative directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each water sample to be collected. Samples collected for the analysis of other metals may be shared for mercury analysis provided a minimum of 0.5-L of aqueous sample is available. Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. See project specific field sampling plan (FSP) for sample collection and preservation instructions. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.

7.4 One 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples in accordance with EPA SW-846 Method 6010B or 7000 series methods. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Provide 1 bottle for each solid sample. Quality control samples such as MS and MSD should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. These samples may be shared with samples collected for other analyses such as metals by ICP (6010B) or AA (7000), chlorinated pesticides (8081A), PCBs (8082), other pesticides (8140, etc.), semivolatile organics (8270C) and diesel range organics (DRO, 8015-mod.) or TPH.

7.5 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Analytical data generated outside of the specified holding times must be considered to be minimum values only. Such data may be used to demonstrate that a waste is hazardous where it shows the concentration of a constituent to be above the regulatory threshold but cannot be used to demonstrate that a waste is not hazardous. The holding times for

the extraction and/or analysis of aqueous and soil samples (including TCLP leachate preparations) are 28-days from the date of sample collection.

8.0 HEALTH AND SAFETY

8.1 Calibration standards and other standards used for quality control contain a number of toxic heavy metals at high concentrations. The toxicity of these complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

8.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

8.3 Reagents used such as acids are highly corrosive. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

8.4 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

8.5 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

9.0 WASTE DISPOSAL

9.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

9.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

9.3 Soil samples that have undergone extraction should be free of contaminants. They can be disposed off into sanitary dumpsters. Dispose of sample extracts containing high levels of acid in accordance with applicable AML SOPs.

10.0 PROCEDURE

10.1 Sample Digestion

10.1.1 Water Sample Digestion. To a 10-mL water sample in a digestion tube, or an aliquot diluted to 10 mL, add reagents as shown below:

a. Add 0.5-mL of conc. H_2SO_4
b. Add 0.25-mL of conc. HNO_3
c. Add 1.5-mL of conc. $KMnO_4$ Solution
d. Add 0.8-mL of conc. $K_2S_2O_8$ Solution
e. cap tightly and mix well.

NOTE: If $KMnO_4$ turns clear (e.g. sewage samples), add more $KMnO_4$ solution (solid, if necessary) until the purple color persists.

Digest for two hours at 95°C in a water bath or oven and allow them to

cool. Set it aside and proceed to Section 10.1.7. Record sample preparation information in laboratory logbooks (see Appendix 1 for details).

10.1.2 Digestion of aqueous calibration standards. Prepare aqueous blanks (calibration blank, ICB, and CCBs) and calibration standards (ICAL, CCVs and ICV) as described in Section 6.12 and 6.16. Then proceed with step 10.1.1 for sample digestion and documentation.

10.1.3 Digestion of aqueous spike samples (LCS/LCSD, MS/MSD). For water samples, the volume of deionized water/sample and standards must equal 10.0-mL. Prepare LCS/LCSD samples as described in section 6.14 and MS/MSD samples as described in section 6.15. Then proceed with step 10.1.1 for sample digestion and documentation.

10.1.4 Soil Sample Digestion. Weigh in 0.6-g (0.6-g to 0.8-g range is preferred) of the soil, sediment or waste sample directly into a digestion tube. Record sample weights to three significant figures (to an accuracy of 0.001 g) in the logbook. Add 5-mL water and 5-mL of aqua regia, cap the vial (watchglass) and let stand for 5-min. at room temperature and 2-min. at 95±3°C in the block digester. Add 50-mL of reagent water and 15-mL of conc. $KMnO_4$ Solution. Mix thoroughly and digest for 30-min. at 95±3°C in the block digester. Remove from the block digester and allow the digestates to cool. Proceed with Section 10.1.7 immediately prior to instrumental analysis. Record sample preparation information in laboratory logbooks (see Appendix 1 for details). NOTE: Perform sample preparation in a hood.

10.1.5 Digestion of soil calibration standards. Prepare calibration blanks and calibration standards as described in Section 6.13 and 6.16. Then proceed with step 10.1.4 for sample digestion and documentation. Do not add 5-mL of water to these samples immediately prior to the addition of 5-mL aqua regia.

10.1.6 Digestion of soil spike samples (LCS/LCSD, MS/MSD). For soil samples, the volume of deionized water/sample and standards must equal 5.0-mL. Prepare LCS/LCSD samples as described in section 6.14 and MS/MSD samples as described in section 6.15. Then proceed with step 10.1.4 for sample digestion and documentation. Do not add 5-mL of water to these samples immediately prior to the addition of 5-mL aqua regia.

10.1.7 Environmental samples, associated quality control and calibration samples are ready for instrumental analysis. The instrument mixes the sample digestates (treated immediately prior to instrumental analysis with hydroxylamine sulfate as described below) with stannous sulfate to produce cold mercury vapor, which are determined by atomic absorption:

10.1.7.1 Aqueous Digestates (from Section 10.1.1) Fortify aqueous digestates (10^{+}-mL) with 0.60-mL of the sodium chloride-hydroxylamine sulfate solution to reduce excess permanganate. Load the vials on the instrument for analysis.

10.1.7.2 Soil Digestates (from Section 10.1.4) Fortify the soil digestate with 6.0-mL of the sodium chloride-hydroxylamine sulfate solution to reduce excess permanganate. Load the vials on the instrument for analysis.

10.2 Instrumental Analysis. This is generally done early in the morning since a run of 40 tubes will take six to seven hours to analyze, including instrument warm-up and stabilization time. The instruments are equipped with containers for acid rinse solutions and the stannous chloride reagent for generating cold mercury vapor from the samples and standards.

10.2.1 Leeman Labs Analyzer – Install and set up the instrument with reservoirs containing the reductant and rinse solutions, rinse cup, and a receptacle for receiving waste from the instrument. Set up the WinHg program, which consists of three separate applications (Runner, Database, and Rack Edit) for instrument setup and control. Instrumental procedures for instrument setup, calibration and sample analysis are described below:

10.2.1.1 Calibrate the autosampler as described in Section 2.10 of the Instrument Manual.

10.2.1.2 Light the Hg lamp by clicking the "ON" button in the "Hg lamp group" from the Runner "Control" tab. Adjust the lamp, if necessary with the "Adjust" button.

10.2.1.3 Test the system, if necessary as described in Section 2.12 of the Instrument Manual.

10.2.1.4 Turn on the peristaltic pump by clicking the "ON" button in the "Pump Group" from the Runner "Control" tab.

10.2.1.5 Turn on the gas flow by clicking the "ON" button in the "Gas Group" from the Runner "Control" tab.

10.2.1.6 The instrument can be run in the low concentration (ppt) or normal (ppb) mode. Use 2% HCl as the rinse in the in the ppt mode. A 10% HCl rinse is used for the normal ppb level measurements.

10.2.1.7 Setup and name a protocol (e.g. "hgppb1") by clicking the "database" button from the Runner Control "Main" tab. Select "File" from the "database", select "New Protocol" and enter the name of the protocol. Click on the appropriate box (e.g. "ppb") under "Protocol type". Click "OK" to set up the new protocol. The WinHg software will select appropriate protocol parameters (e.g. sample introduction, pump times, replicates, integration times, etc.) for the protocol type (ppt, ppb, ppm, or CLP) selected. Check the "Weight" and "volume" boxes under the "Calculations" group so that sample specific weight and/or volumes can be entered in the Autosampler table. Use the tab key to move between the fields.

10.2.1.8 Select the "Line Info" tab and enter the concentrations of the "Calibration stds" (for ICAL), "Check stds" (for ICVs, CCVs,

ICBs, CCBs), "Units" (e.g. ppb). Click on the "Apply" button to save the data. Do not enter standard concentration data under the "Standard Addition" data unless the MSA procedure is performed.

10.2.1.9 **NOTE:** MSA is a sample-specific calibration and quantitation procedure. To perform MSA, the "Analysis Type" on the "Protocol" tab should be changed to "Methods of Standard Additions" and the concentration data for the "Standard additions" should be entered.

10.2.1.10 The WinHg software is capable of generating real time (while samples are getting analyzed) and post-run reports. Select the desired items to appear in the reports by clicking on the "report" tab in the "database". Click on the "RN" button on the "database" toolbar to exit from this program.

10.2.1.11 Creating Data Set – This procedure is used to create a file or "dataset" for storing data. For example, create a file by name "#ALPHA" by clicking on "file" on the Runner's pull down menu, selecting "New data Set" and entering "#ALPHA". Including the "#" sign in the filename will allow data from any protocol to be saved to this data set. Enter a batch name by selecting the "Start New Batch" and entering a batch name (e.g. "WO-4206" or "05Jan2004").

10.2.1.12 Autosampler Setup – The autosampler has three stations: Station-1, which has 14 positions for standards; and Station-2 and Station-3 with each having 44 positions for samples. Launch the "Rack Edit" application by clicking on the Autosampler rack icon on the toolbar or the "Rack Editor" button on the Runner "main" tab. Load the aqueous standards (S1=0, S2=0.2, S3=1.0, S4=2.0, S5=5.0, S6=10.0 ppb) in positions 1 to 6 of Rack-0. Load the samples in Station-1 (and Station-2, if necessary) of the autosampler cups. Enter the AML standard name and the AML sample numbers on the "Sample ID" tab corresponding to the sample cup. Enter weights and volumes as appropriate.

10.2.1.13 Click on the "Standard" tab. The "Active Cups" map illustrates the positions that are occupied. Unoccupied positions will be grayed out in the screen. Select the standards to be used fir initial calibration by clicking on the buttons for S1, S2, S3, S4, S5 and S6. Set the number of replicates to three by clicking on the "Rep 3" button. Click on the "real time print" check box and click on the "Stnd Auto" button. The instrument will analyze the standards and construct a calibration curve using the mean of three replicates for each standard.

10.2.1.14 Reviewing the Initial Calibration Data – Click on the "DB" button on the toolbar and click on the "Cal curve" tab. The instrument will display the calibration curve, regression parameters (e.g. a, b and c), correlation coefficient ("rho"), observed

intensities for each replicate and their mean for each standard, the nominal value for each standard and the calculated value from the calibration curve. If the correlation coefficient is >0.995 and the deviations for the calculated values for each level are low, accept the ICAL by clicking the "Accept" button.

10.2.1.15 Sample Analysis – Click on the "RN" button from the database application for returning to the WinHg Runner. Click on the "Sample" tab. Select "Station 1" under the "Autosampler run" group and enter the starting and ending cups for samples that are loaded in Station-1. Click the "Run Auto" button. When the "Output" tab pops up, click on the "Report" tab.

10.2.1.16 Report Generation General– This utility allows the selection of specific data for reports. It is important that 'Data Sets" and "batches" are selected correctly before the sample are analyzed. Samples for each AML work order may be segregated by using appropriate work order number for batches. Samples from multiple AML work orders, standards (ICAL, ICV, ICB, CCVs, CCBs, FCV, and FCB) and associated batch QC samples (MB, LCS, LCSD, MS, MSD, SD, PDS, etc.) may be segregated from other QC batches by employing preparation batch numbers for the name of dataset.

10.2.1.17 Report Generation Procedure – Return to the WinHg Runner, click on the "DB" button, click on the "Report" tab, select "water" in the "Report Spec" group for aqueous samples, click on the "In" box for the batch to be reported in the "Batch List" group, and click on the "Generate Report" button in the lower right corner of the screen. When the "Report Generation" menu pops up, select the "viewer only" to view data on the screen prior to selecting the "printer" for generating hardcopy reports.

10.2.2 Spectro Products Analyzer – This instrument is used only as a back-up instrument for CVAA mercury analysis.

10.2.2.1 First turn on the Hg analyzer, set ATTN. To 1x (middle switch) and let warm up at least 30 minutes. Adjust baseline to 100 with B setting in back. Turn on recorder. Start stirring $SnCl_2$ and make up more $SnCl_2$ if the container is less than one-half full. Adjust B to 100again if the pointer has drifted. If AGC is not 100, adjust screw control in the back of the instrument so it is 100. Then adjust A so baseline is near zero. Let stabilize on 10x attenuation while reducing samples in step 10.3. Instrument stabilization usually takes 30 minutes. Adjust baseline with A before running samples and with recorder after starting samples.

10.2.2.2 Reducing sample digestates. Cool samples in cool water until samples are at room temperature. Add 0.6 mL of 12% NH_2OH-HCl solution to each tube, tighten cap, and invert tube several times over a period of 15 minutes. When all brown

permanganate color is gone, samples are ready to analyze.

10.2.2.3 Running Samples. Set up Chemstation as described in the instrument manual. When the baseline is steady and samples are reduced with NH_2OH, put samples into sample tray using same test tubes as for digesting. Fill the tray keeping only about one hour ahead of sample run (10 samples). This limits the exposure to the air and ensures that the sample does not have time to stratify after mixing. Run reagents only for an initial good baseline before actually running samples. Monitor the peaks for the standards, spikes, etc. on the screen. Perform serial dilution, standard addition and post digestion spike analyses (when applicable) as described in the quality control section.

10.2.2.4 Turning off Equipment. After all samples are finished, the pump tubes need to be washed out with deionized water and the $SnCl_2$ tube may need to be washed out with acid rinse. Turn off the recorder. Turn off analyzer by putting A and B pots in their counterclockwise stop positions. Turn off other equipment and take the pump tube cover off the pump tubes. Replace the pump tubes when peaks on recorder becomes shorter and fatter.

10.2.2.5 Cleaning Test Tubes. Wash each tube with tap water and then deionized water. Place in the test tube rack with caps on loosely. Heat in oven at 95°C for at least four hours.

10.2.2.6 Instrument Response - Peak areas for each sample and/or standard should be documented in project files and Excel spreadsheets (see Appendix 2 and 3 for details).

10.2.2.7 Calibration curve. Using Excel spreadsheets, process the soil and/or water analytical data to calculate calibration factors, mean calibration factors, linear regression parameters, percent difference for ICVs, CCVs, etc. (see Appendix 3 for details). The correlation coefficient for linear regression should be greater than or equal to 0.995.

10.2.2.8 Calculations. Perform calculations using Excel spreadsheets (see Appendix 3) to determine sample amounts and spike recoveries for LCS, LCSD, MS and MSD.

10.3 Data review and verification. Organize data and review standard and sample results using a checklist (see Appendix 4 for details).

10.4 Reporting. Report sample results in accordance with applicable method detection limits (MDL), laboratory reporting limits (RL) and method quantitation limits (MQL) as shown in Tables 3 and 4. Use Form-1 equivalents (see Appendix 9 for details) to report aqueous and solid sample results.

10.5 Processing Quality Control Results. Compare spike recoveries for laboratory control samples and matrix spikes with quality control limits as shown in Tables 3 and 4 for aqueous and soil/sediment samples respectively.

11.0 QUALITY CONTROL

11.1 Quality control (QC) procedures are used to ensure that proper sample preparation technique, extract cleanup procedures, and instrument calibration procedures are performed. This involves setting up multi-point initial calibration, initial verification of the calibration (ICV), continuing verifications of the calibration during the analysis sequence (CCVs), and the final verification of the calibration at the end of sample analysis (FCV). It also involves the verification of calibration blanks throughout the analytical sequence (ICB, CCBs and FCB) and the analysis of method blanks (MB), laboratory control samples (LCS and LCSD), and matrix spikes (MS and MSD).

11.2 Establish method detection limit (see Appendix 8 for details), reporting limit (RL), and the practical quantitation limit (MQL). See Tables 3 and 4 for summary.

11.3 Initial Calibration - Analyze a calibration blank and five levels of calibration solutions that were digested using the same procedure as the samples. Plot instrument response against the calibration standard concentration on new Excel spreadsheet for each batch of samples analyzed. The correlation coefficient for the initial calibration (ICAL) should be equal to or greater than 0.995 for the calibration to be acceptable.

11.4 ICV- initial calibration verification (ICV) must be performed after instrument calibration procedures have been completed. The ICV should be within ±10% of the true value. If reanalysis of the ICV is unsatisfactory, recalibration is required. If the problem persists the analysis is halted and instrument maintenance is performed. Document ICV on new Excel spreadsheets for each batch of samples analyzed.

11.5 CCV/FCV- The continuing calibration verification (CCV) and final calibration verification (FCV) check standard sample is analyzed every 10th sample and at the end of sequence respectively. They should be within ±20% of the true value. Otherwise, data for the previous ten samples may be considered unusable (or estimated depending on the monitoring program) and must be reanalyzed. Document CCV/FCV on new Excel spreadsheets for each batch of samples analyzed.

11.6 ICB/CCB/FCB - An initial calibration blank (ICB) is analyzed to verify that no target analytes have been inadvertently introduced into the calibration reagents. ICB analysis must be performed after instrument calibration procedures have been completed. The continuing calibration blanks (CCBs) and final calibration blank (FCB) are analyzed every 10th sample and at the end of sequence respectively. The calibration blanks follow the analysis of calibration verification in the run sequence. The default calibration blank criteria is < MDL that is applicable to the matrix under consideration. If reanalysis of the calibration blanks are unsatisfactory, recalibration is required. If the problem persists the analysis is halted and the source of contamination determined. Document ICB/CCB/FCB on new Excel spreadsheets for each batch of samples analyzed.

11.7 The frequency of analysis of LCS analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples. LCS can be run in duplicate to obtain precision (RPD) information. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. LCS recoveries should

be within ±20% of the true values. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. LCS can be run in duplicate to obtain precision (RPD) information. Report LCS/LCSD results in Form-1 equivalents and recoveries/RPD in Form-3 equivalents (see Appendix 5 for details).

11.8 The frequency of analysis of MS analysis is equivalent to a minimum of 2 per 20 samples or 2 per batch if less than 20 samples. The MS recoveries should be within ±20% of the true values. MS is run in duplicate to obtain precision (RPD) information. The RPD for duplicates, which includes MS duplicates, should be within 20%. Report MS/MSD results using Form-1 equivalents and recoveries/RPD using Form-3 equivalents (see Appendix 6 for details).

11.9 The frequency of analysis of MB analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples.

11.10 Post digestion spike (PDS) analysis - Perform post digestion spike using the mid-level or other appropriate spiking level as shown in Table 2. The PDS recovery should be within ±15% of the true value.

11.11 Serial Dilution Procedure – Perform 1:5 dilution of one sample from each analytical batch and compare the results for the two analysis. Samples selected for matrix spike analysis can be selected for routine performance of this test. If the results agree within ±10%, the absence of interferences is indicated. Perform serial dilution test on another sample, if the original (undiluted) value is less than 10 times the MDL. Perform recovery test if all the sample results are less than 10 times the MDL.

11.12 Method of Standard Additions (MSA) – This procedure should be used when interferences are detected from dilution and recovery tests performed as described above. A known volume of the sample extract (V_x) is spiked with a known volume (V_s) of a standard solution of known concentration (C_s) so that the spiked sample concentration (S_a) is approximately twice the concentration of the unspiked sample (S_b). The unspiked sample is diluted with the same volume (V_s) of reagent water. Both samples are analyzed on GFAA and the unknown sample concentration (C_x) is calculated using the following equation:

$$C_x = \frac{S_b * V_s * C_s}{(S_a - S_b) * V_x}$$

where
V_x = Volume of sample extract
V_s = Volume of standard solution spiked
S_a = Measured concentration of spiked sample
S_b = Measured concentration of the unspiked (diluted) sample
C_s = Concentration of the spiking standard
C_x = Concentration of the unknown sample to be reported

11.13 The MSA can be performed using multiple spikes and the C_x calculated graphically from a plot of the response against the concentration of the standard added to the sample digestate (see Appendix 10 for details). Use at least 3 spiking levels at

approximately 50%, 100%, and 150% of the original concentration (see Table 2 for MSA scheme) in addition to the analysis of the diluted sample. The spiked and unspiked samples should contain the same amount of sample extract diluted to the same final volume as shown in Table 2. The calculations can be performed using Excel spreadsheets.

11.14 Any sample that has an element response above the established linearity of the instrument is diluted and reanalyzed.

11.15

11.16 Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

12.0 **DATA VERIFICATION AND VALIDATION -** This section will provide guidance and procedures to assess if data produced using this SOP are valid and legally defensible. Document review and comments on the checklist (See Appendix 4 for details). Verify that sample preparation logs are complete and the instrument run logs are available for review (See Appendix 1 for details). Anomalies and exceptions should be documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on the checklists. Army (USACE) and Air Force (AFCEE) projects may have special requirements (See Appendix 11) that will require extra attention. A simple, universal procedure for evaluating and choosing appropriate corrective action doe not exist to deal with situations when one or more QC parameters are outside the acceptance window. The objective of the laboratory is to provide "data of known quality" that meets client objectives, which includes project data quality objectives as well as cost and schedule constraints.

12.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) or air force (AFCEE) projects may have additional requirements that are related to USACE laboratory validation (CX), USACE District requirements, government contract requirements, specific project requirements or Architect/Engineer (A/E) requirements. Contact supervisor for specific client requirements (see Appendix 11 for details).

12.1.1 The USACE and/or AFCEE requirements (see Appendix 11 for details) and elsewhere in this SOP are client specifications, which are based on their understanding of method quality objectives (MQOs), which when not met, do not affect the legal validity of data. Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may or may not be attainable for routine sample analysis. Since MQOs are simply objectives that may not always be attainable by commercial laboratories operating under schedule and cost constraints, client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC

problems as they arise. Quality Assurance Project Plans (QAPPs), when available, should guide necessary corrective action.

12.1.2 The laboratory should strive to generate data of known quality meeting referenced method requirements within the constraints of project schedule and cost. Samples run under rapid turnaround conditions (one week or less) may require reporting of results that may not meet all method quality objectives and/or other QC requirements. Exceptions and data qualifications necessary should be documented on the checklists and eventually on case narratives.

12.1.3 For Level 1 Analyst Review, the analyst should assemble records as stated on the checklist, perform review as stated in this Section of the SOP, and initial/date as "Reviewer 1", when the analyst review is complete.

12.1.4 For Level 2 Peer Review, another analyst, supervisor, or data review specialist will review the checklist and the data package, performing independent checks of the analyst's conclusions, recording additional comments (as and if necessary) and initial/date as "Reviewer 2", when the review is complete.

12.1.5 For Level 3 Management/Reporting/QA Review, the Operations Manager, QA Manager, QA Director or the Laboratory Director will review the checklists (initial/date as necessary), prepare case narrative (if not already prepared by the report generation team), and review the final report package for errors, omissions, non-conformance, etc.

12.2 HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

12.2.1 WATER - The holding times for sample digestion and analysis is 28 days from sampling. Water samples include TCLP leachates.

12.2.2 SOIL – The holding times for sample digestion and analysis is 28 days from sampling.

12.2.3 WASTE - The holding times for TCLP leachate preparation is 28 days from sampling.

12.2.4 Holding times are established by comparing the chain of custody sampling date with the date of the injection.

12.2.5 ACTION – If holding times have been exceeded, contact the client. All positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or invalid (I).

12.3 INITIAL CALIBRATION VERIFICATION (ICAL and ICV) – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the instrument is capable of producing data within a range that is linear. ICV demonstrates that

instrument performance has not changed since the generation of ICAL.

12.3.1 Element – Check to see that the correlation coefficient (r) for linear regression is greater than 0.995. Check the lowest calibration point for deviations greater than 20% from true value to establish method or practical quantitation limit (MQL) for the analysis.

12.3.2 ACTION – If r is <0.995, correct the problem and reanalyze calibration solutions. If the lowest calibration point is >± 20% from true value, use the next higher point to compute PQL, provided this point satisfies the criteria. The MDLs, RLs, and PQLs are shown in Tables 3 and 4 for aqueous and soil matrices.

12.3.3 Element – Check to see that ICV is within ± 10% of the true value. If QC criteria is not satisfied, correct the problem and reanalyze ICV (and ICAL calibration solutions, if necessary) for an acceptable ICV.

12.3.4 ACTION – Contact client if corrective action does not resolve problem within the schedule for the project. Positive results for compounds not meeting the criteria may need to be qualified as estimated (J) by the user (client). Report detects and non-detects without qualifiers and mention these outliners in the case narrative.

12.4CONTINUING CALIBRATION (CCVs and FCV)– The continuing and final calibration verification solutions are used to check that the instrument is still operating under the linear range of the initial calibration and has remained stable over the entire analytical sequence.

12.4.1 CCVs and FCV values should not deviate more than ± 20% of the true value.

12.4.2 ACTION – If QC criteria is not satisfied, correct the problem and reanalyze CCVs and FCV and all associated samples. Associates samples include all samples that precede and follow the affected CCV. Contact client if corrective action does not resolve problem within the schedule for the project. Positive results for compounds not meeting the criteria may be qualified as estimated (J) by the user (client). Report non-detects without qualifiers and mention these outliners in the case narrative.

12.5METHOD BLANKS and CALBRATION BLANKS (ICB, CCBs, and FCB) – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Sample data qualification to alert blank contamination is performed by the laboratory. Sample data qualification by the client based on all the blank results (laboratory MB, calibration blanks, equipment blank, and other field blanks) is performed by the user (client). The processing of blanks helps eliminate reporting of false positives in project reports.

12.5.1 Method blanks and calibration blanks should be free of all target analytes

and other interfering non-target analytes. To be acceptable, blank results should not exceed one half of the MQL. For some projects, it cannot exceed ±MDL.

12.5.2 Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples. If levels greater than one half the PQLs are present, inspect and correct the problem. Reanalyze all associated samples. Contact client if other criteria are applicable. The only exception may be quick turn around projects.

12.5.3 ACTION – All positive results are reported with a "B" code, if contaminants are found in the method blank.

12.5.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, then no action is taken.

12.5.5 ACTION – Any compound detected in the sample which were also detected in associated method blank at levels greater than one half the PQLs should be re-digested unless other client criteria are applicable.

12.5.6 ACTION – If gross contamination is observed, all affected elements are qualified as invalid (I). Redigestion and/or reanalysis will be necessary.

12.6 LABORATORY CONTROL STANDARD (LCS) – A LCS analysis is used to evaluate method competence in the absence of matrix effects. The LCS should be from a second source standard. The LCS may provide a mechanism to check method efficiency for target compounds without the sample matrix as a factor.

12.6.1 Document LCS and/or LCSD results in Form-1 equivalents and percent recoveries/RPD in Form-3 equivalents (see Appendix 5 for details)

12.6.2 Check the results for the percent LCS recoveries to see if the results are within the acceptable QC limits (80 to 120%) for water and soil. The RPD should be within QC limits (<20%) for water and soil. Flag outliers with an asterisk (*).

12.6.3 ACTION – No action is taken on LCS recovery data alone to qualify all associated samples. The supervisor should be consulted if this is not achievable. If significant deviations are noticed, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements, which may be compound specific. Check with the project manager about such requirements. No action may need to be taken on LCS and/or LCSD recovery data alone either to reanalyze samples and/or to qualify associated samples for most projects. Professional judgment may be used by the user in evaluating sample data qualification options in conjunction with other QC data for the project.

12.7 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike data are used to evaluate the precision and accuracy of the analytical method on project samples.

12.7.1 Report MS/MSD results in Form-1 equivalents and percent

recoveries/RPD in Form-3 equivalents (see Appendix 6 for details) and 8.

12.7.2 Check the results for percent MS/MSD recoveries and compare them to the QC limits for water and soil (80-120). The RPD should be within QC limits for water and soil (20). Flag outliers with an asterisk (*).

12.7.3 Perform one manual calculation using raw data and extraction sheet information.

12.7.4 ACTION – No action is taken on MS/MSD recoveries data alone to qualify all associated samples. Since matrix interference could lead to lead to serious analytical problems, the supervisor should be consulted when significant outliers are noted. Document QC outliers for %recovery and RPD on the checklist and on client reports (*) and draw attention to the QC outliers on the case narrative. No action may be taken on MS/MSD recovery data alone either to reanalyze samples and/or to qualify associated samples either by the analyst or by the end user. Professional judgment may be used by the user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

12.8 SAMPLE DUPLICATES – Laboratory duplicate analysis can be performed instead of analyzing Matrix Spike (MS) in duplicate. The relative percent difference (RPD) between the duplicate results is used to evaluate the precision of the analytical method on project samples.

12.8.1 Document sample duplicate results in Form-3 equivalents (see Appendix 5 and 6 for details). Calculate RPD as described for LCS/LCSD and/or MS/MSD and check the observed RPD against QC limit. The RPD should be within QC limits for water (<20%) and soil (<20%). Flag outliers with an asterisk (*).

12.8.2 ACTION – No action is taken on RPD data for soil samples since matrix interferences and inhomogeneity may cause problems. If the LCS is run in duplicate, check the RPD for laboratory control of precision. Document QC outliers for RPD on the checklist and on client reports (*) and draw attention to the QC outliers on the case narrative. Professional judgment may be used by the user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

12.9 RESULTS – Report sample amounts that are greater than the reporting limit (RL) or method detection limit (MDL) using "J" qualifiers if the levels are less than PQL. Positive sample results associated with positive MB results should also be qualified with a "B" code. Report soil and water results using Form-1 equivalents (see Appendix 9 for details).

12.9.1 If the concentration exceeds the upper limit of calibration (ICAL), flag the result with an estimated or "E" flag. The extracts may need to be diluted and reanalyzed until the concentrations are within the calibration limits for

normal turnaround analyses.

12.9.2 The lowest calibration point is used as PQL for aqueous sample analysis. However, due to matrix interference, the lowest calibration point is used as the reporting limit for soil and the next higher level is used as PQL.

13.0 **REFERENCES**

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13.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 7471A, Mercury in Solid 0r Semisolid Waste (Manual Cold-Vapor Technique), September 1994.

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13.18 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.
13.19 AML Standard Operating Procedures, Q-Water-RC.
13.20 AML Standard Operating Procedures, Q-Subsampling-RC.
13.21 AML Standard Operating Procedures, Q-WasteMgmt-RD.
13.22 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
13.23 AML Standard Operating Procedures, Q-Controlcharts-RC.

Table 1

SAMPLE VOLUMES AND HOLDING TIMES

Measurement	Digested Volume (mL)	Collection Volume (mL)	Holding Time Preservation
Total	10	500	28 days, HNO3 to pH <2
Dissolved	10	500	28 days, filter on site; HNO3 to pH <2
Suspended	10	500	28 days, filter on site
TCLP-Mercury	10	500	28 days
Solid (Total)	0.6-g	100-g	28 days

Table 2

Dilution Scheme for Method of Standard Addition

Volume of Sample Extract (mL)	Volume of Spiking Solution (mL)	Volume of Reagent Water (mL)	Final Volume of Diluted Extract (mL)
5.0	0.0	5.0	10.0
5.0	1.0	4.0	10.0
5.0	2.0	3.0	10.0
5.0	3.0	4.0	10.0
5.0	5.0	0.0	10.0

Table 3
Soil/sediment Sample MDL, MQL, and QC Limits for LCS and MS

Metals Analysis by Atomic Absorption Spectroscopy (AA)

Analytical Management Laboratories

Matrix: Water	**Sample Preparation**		**Sample Analysis**			**Technique**
Element	Method No.		Method No.		Wavelength (nm)	
Mercury	7470A		7470A		253.7	Cold Vapor (CVAA)

	Spike Amount	Mean (B)	S.D.	**MDL (A)**	Ratio (RD)		**MQL (C)**	**Ratio (RQ)**		**LCS Limits %R**		**MS Limits %R**		**RPD**
Element	(ug/L)	ug/L	ug/L	**ug/L**	RD= B/A	flag	**ug/L**	RQ= C/A	flag	Lower	Upper	Lower	Upper	Limit
Mercury	0.25	0.26	0.024	**0.077**	3.4		0.50	6.5		80	120	80	120	20

Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 5.

Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.

LCS and MS %R limits are from the USACE EM200-1-3, Appendix I.

Table 4
Aqueous Sample MDL, MQL, and QC Limits for LCS and MS

Metals Analysis by Atomic Absorption Spectroscopy (AA)

Analytical Management Laboratories

Matrix: Soil	**Sample Preparation**		**Sample Analysis**			**Technique**
Element	Method No.	Date	Method No.	Date	Wavelength	
Mercury	7471A/7471B		7471A/7471 B		253.7	Cold Vapor (CVAA)

	Spike Amount	Mean (B)	S.D.	**MDL (A)**	Ratio (RD)		**MQL (C)**	**Ratio (RQ)**		**LCS Limits %R**		**MS Limits %R**		**RPD %**
Element	(ug/kg)	ug/kg	ug/kg	**ug/kg**	RD= B/A	flag	**ug/kg**	RQ= C/A	Flag	Lower	Upper	Lower	Upper	Limit
Mercury	20.8	26.0	4.73	**14.9**	1.7		41.7	2.8	*	80	120	80	120	20

Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 10.

Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.

LCS and MS %R limits are from the USACE EM200-1-3, Appendix I.

Table 5
List of Appendices

Appendix Number	Appendix Topic
1	Mercury Sample Digestion Logbook Sheets (Soil and Water) Instrument Run Logs
2	Instrument Printouts (Leeman Labs & Spectro Analyzer)
3	Excel Spreadsheets for internal data processing Initial Calibration (ICAL) Summary (Form-6 equivalent benchsheets) Calibration Verification Summary (Form-7 equivalent benchsheets) Sample Results Summary (Form-1 equivalent benchsheets) Surrogate, LCS and MS Summary (Form-2 and 3 equivalent benchsheets)
4	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
5	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
6	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
7	QC Association Forms (Form-4 equivalents)
8	Method Detection Limit (MDL) Study Summary
9	Sample Reports (Form-1 equivalents)
10	Method of standard Addition (MSA)
11	Client Method Quality Objectives for Method 7470A and 7471A (AFCEE, USACE, DoD QSM, etc.)

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. I-Acidity-RC

Acidity by Titrimetry
(EPA 305.1 and SM 2310B)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

ACIDITY by TIREIMETRY (EPA 305.1 and SM 2310B)

1.0 Scope and Application

1.1 This SOP is based on EPA Method 305.1, 305.2, and Standard method 2310B for the determination of acidity by titration.
1.2 This method is applicable to surface waters, sewage and industrial wastes, particularly mine drainage and receiving streams, and other waters containing ferrous iron or other polyvalent cations in a reduced state.
1.3 This method covers the range from approximately 10 mg/L acidity to approximately 1000 mg/L as $CaCO_3$, using a 50 mL sample.

2.0 Summary of Method

2.1 The pH of the sample is determined and a measured amount of standard acid is added, as needed, to lower the pH to 4 or less. Hydrogen peroxide is added, the solution boiled for several minutes, cooled. And titrated electrometrically with standard alkali to pH 8.2.

3.0 Definitions

3.1 This method measures the mineral acidity of a sample plus the acidity resulting from oxidation and hydrolysis of polyvalent cations, including salts of iron and aluminum.

4.0 Interferences

4.1 Suspended matter present in the sample, or precipitates formed during the titration may cause a sluggish electrode response. This may be offset by allowing a 15-20 second pause between additions of titrant or by slow drop-wise addition of titrant as the endpoint pH is approached.

5.0 Apparatus

5.1 pH meter, suitable for electrometric titrations.

6.0 Reagents

6.1 *Hydrogen peroxide*, (H_2O_2, 30% solution)
6.2 *Standard Sodium Hydroxide,* 0.02 N.
6.3 *Standard sulfuric acid,* 0.02 N.

7.0 Procedure

7.1 Pipet 50 mL of the sample into a 250 mL beaker.
7.2 measure the pH of the sample. If the pH is above 4.0, add standard sulfuric acid (6.3) in 5-mL increments to lower the pH to 4.0 or less. If the initial pH of the sample is less than 4.0, the incremental addition of sulfuric acid is not required.
7.3 Add 5 drops of hydrogen peroxide (6.1)
7.4 Heat the sample to boiling and continue boiling for 2 to 4 minutes. In some instances, the concentration of ferrous iron in a sample is such that an additional amount of hydrogen peroxide and slightly longer boiling time may be required.
7.5 Cool the sample to room temperature and titrate electrometrically with standard sodium hydroxide (6.2) to pH 8.2.

8.0 Calculations

8.1 Acidity, as mg/L $CaCO_3$ = $\frac{[(A * B) - (C * D)] * 50{,}000}{\text{mL of sample}}$

where

A = volume of standard sodium hydroxide used in titration
B = normality of standard sodium hydroxide
C = volume of standard sulfuric acid used to reach pH to 4 or less
D = normality of standard sulfuric acid

8.2 If it is desired to report acidity in millequivalents per Liter, the reported values as CaCO3 are divided by 50 as follows:

$$\text{Acidity as meq/L} = \frac{\text{mg/L } CaCO_3}{50}$$

9.0 References

9.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Acidity, Method 305.1, Titrimetric, Technical Revision, 1974.
9.2 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Acidity, Method 305.2, Titrimetric, December 1982.
9.3 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Acidity, Method 2310A and 2310B.
9.4 AML Standard Operating Procedures, Q-Water-RC.
9.5 AML Standard Operating Procedures, Q-WasteMgmt-RD.
9.6 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
9.7 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-Alkalinity-RC

Alkalinity by Titrimetry
(EPA 310.1)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ __ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

ALKALINITY (EPA METHOD 310.1, Titrimetric, pH 4.5)

1.0 Scope and Application

1.1 This method is applicable to drinking, surface and saline waters, domestic and industrial wastes.
1.2 The method is suitable for all concentration ranges of alkalinity; however, appropriate aliquots should be used to avoid a titration volume greater to 50 mL.
1.3 Automated titrimetric analysis is equivalent.

2.0 Summary of Method

2.1 An unaltered sample is titrated to an electrometrically determined end point of pH 4.5. The sample must not be filtered, diluted concentrated or altered in any way.

3.0 Sample Preservation and Comments

3.1 The sample should be refrigerated at 4 ^{o}C and run as soon as practical. Do not open sample bottle before analysis.
3.2 Substances, such as salts or weak organic and inorganic acids present in large amounts, may cause interference in the electrometric pH measurements.
3.3 For samples having high concentrations of mineral acids, such as mine wastes and associated receiving waters, titrate to an electrometric endpoing of pH 3.9, using the procedure in: Annual Book of ASTM Standards, Part 31, "Water", p 115, D-1067, method D, (1976).
3.4 Oil and grease, by coating the pH electrode, may also interfere, causing sluggish response.

4.0 Apparatus

4.1 pH meter or electrically operated titrator that uses a glass electrode and can be read to 0.05 pH units. Standardize and calibrate according to manufacturer's instructions. If automatic temperature compensations is not provided, make titration of 25 ± 2°C.
4.2 Use an appropriate sized vessel to keep the air space above the solution at a minimum. Use a rubber stopper fitted with holes for the glass electrode, reference electrode (or combination electrode) and buret.
4.3 Magnetic stirrer, pipets, flasks and other standard laboratory equipment.
4.4 Burets, Pyrex 50, 25 and 10 mL.

5.0 Reagents

5.1 *Sodium carbonate solution,* approximately 0.05 N: Place 2.5 g (to nearest mg) Na2Co3 (dried at 250°C for 4 hours and cooled in desiccator) into a 1 liter volumetric flask and dilute to the mark.

5.2 *Standard acid (sulfuric or hydrochloric)*, 0.1 N: Dilute 3.0 mL conc H2SO4 or 8.3 mL conc HCl to 1 liter with distilled water. Standardize versus 40.0 mL of 0.05 N Na2CO3 solution with about 60 mL distilled water by titrating potentiometrically to pH of about 5. Lift electrode and rinse in to beaker. Boil solution gently for 3-5 minutes under a watch glass cover. Cool to room temperature. Rinse cover glass into beaker. Continue titration to the pH inflection point. Calculate normality using:

$$N = \frac{A * B}{53.0 * C}$$

where:

A = g Na_2CO_3 weighed into 1 Liter
B = mL Na_2CO_3 solution
C = mL acid used to inflection point

5.3 *Standard acid (sulfuric or hydrochloric), 0.02 N*: Dilute 200 mL of 0.10 N standard acid to 1 liter with distilled water. Standardize by potentiometric titration of 15.0 mL 0.05 N Na_2CO_3 solution as above.

6.0 Procedure

6.1 Sample size
- 6.1.1 Use a sufficiently large volume of titrant (>20mL in a 50 mL buret) to obtain good precision while keeping volume low enough to permit sharp end point.
- 6.1.2 For <1000 mg $CaCO_3$/L use 0.2 N titrant.
- 6.1.3 For <1000 mg $CaCO_3$/L use 0.1 N titrant.
- 6.1.4 A preliminary titration is helpful.

6.2 Potentiometric titration
- 6.2.1 Place sample in flask by pipetting with pipte tip mear bottom of flask.
- 6.2.2 Measure pH of sample
- 6.2.3 Add standard acid (5.2 or 5.3), being careful to stir thoroughly but gently to allow needle to obtain equilibrium.
- 6.2.4 Titrate to pH 4.5. Record volume of titrant.

6.3 Potentiometric titration of low alkalinity
- 6.3.1 For alkalinity of <20 mg/L titrate 100-200 mL as above (6.2) using a 10 mL microburet and 0.02 N acid solution (5.3)
- 6.3.2 Stop titration at pH in range of 4.3 - 4.7, record volume and exact pH. Very carefully add titrant to lower pH exactly 0.3 pH units and record volume.

7.0 Calculation

7.1 Potentiometric titration to pH 4.5

$$\text{Alkalinity, mg/L } CaCO_3 = \underline{A * N * 50{,}000}$$

mL of sample

where:

A = mL standard acid
N = normality standard acid

7.2 Potentiometric titration of low alkalinity:

$$\text{Total alkalinity, mg/L } CaCO_3 = \frac{(2B - C) * N * 50{,}000}{\text{mL of sample}}$$

where:

B =mL titrated to first record pH
C = total ml titrant to reach pH 0.3 units lower
N = normality standard acid

8.0 References

8.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Alkalinity, Method 310.1, Titrimetric.
8.2 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998.
8.3 AML Standard Operating Procedures, Q-Water-RC.
8.4 AML Standard Operating Procedures, Q-WasteMgmt-RD.
8.5 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
8.6 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-Ammonia-ISE-RC

Potentiometric Measurement of Ammonia (EPA 350.3 and SM 4500-NH_3-D)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Potentiometric Measurement of Ammonia (EPA 350.3 and SM 4500-NH_3-D)

1.0 SCOPE AND APPLICATION

1.1 This SOP is based on EPA and Standard Method distillation procedures (EPA 350.2 and Standard Method 4500-NH_3-B) and potentiometric measurement (EPA 350.3 and Standard Method 4500-NH_3-D) procedures.
1.2 This SOP covers the determination of ammonia-nitrogen exclusive of total Kjeldahl nitrogen, in drinking, surface and saline waters, domestic and industrial wastes.
1.3 It is the method of choice where economics and sample load do not warrant the use of automated equipment.
1.4 The method covers the range from about 0.03 to 1400-mg NH_3-N/L
1.5 Color and turbidity have no effect on the measurements, thus, distillation may not be necessary.

2.0 SUMMARY OF METHOD

2.1 Distillation is optional for this procedure. For distillation, the sample is buffered at a pH of 9.5 with a borate buffer in order to decrease hydrolysis of cyanates and organic nitrogen compounds, and is then distilled into a solution of boric acid.
2.2 The ammonia in the distillate or the original sample is determined potentiometrically using an ion selective electrode (ISE) and a pH meter having an expanded millivolt scale.
2.3 The ammonia electrode uses a hydrophobic gas-permeable membrane to separate the sample solution from an ammonium chloride internal solution. Ammonia in the sample diffuses through the membrane and alters the pH of the internal solution, which is sensed by a pH electrode. The constant level of chloride in the internal solution is sensed by a chloride selective ion electrode, which acts as the reference lectrode.

3.0 SAMPLE HANDLING AND PRESERVATION

3.1 Samples may be preserved with 2-mL of conc. H_2SO_4 per liter and stored at 4 ^oC.
3.2 Mercuric chloride should not be used to preserve the sample.

4.0 INTERFERENCES

4.1 A number of aromatic and aliphatic volatile amines act as a positive interference.
4.2 When distillation is performed, cyanate, which may be encountered in certain industrial effluents, will hydrolyze to some extent even at the pH of 9.5 at which

distillation is carried out. Residual chlorine must also be removed by pretreatment of the sample with sodium thiosulfate before distillation.

4.3 Mercury interferes by forming a strong complex with ammonia. The sample should not be preserved with mercuric chloride.

5.0 APPARATUS

5.1 An all-glass distilling apparatus with an 80-100 mL flask.
5.2 Erlenmeyer flasks: The distillate is collected in 125-mL flasks. These flasks should be marked at the 25 and 50-mL volumes. With such markings, it is not necessary to transfer the distillate to volumetric flasks.
5.3 Electrometer (pH meter) with expanded millivolt scale.
5.4 Ammonia selective electrode.
5.5 Magnetic stirrer with PTFE coated stirring bar.

6.0 REAGENTS AND CHEMICALS

6.1 Distilled water should be free of ammonia. Such water is best prepared by passage through an ion exchange column containing a strongly acidic cation exchange resin mixed with a strongly basic anion exchange resin. Regeneration of the column should be carried out according to the manufacturer's instructions. NOTE: All solutions should be made with ammonia-free water.
6.2 Ammonium chloride, stock solution: 1.0mL = 1.0 mg NH_3-N. Dissolve 3.819 g NH_4Cl in distilled water and bring to volume in a 1-Liter volumetric flask.
6.3 Ammonium chloride, standard solution: 1.0 mL - 0.01mg. Dilute 10.0 mL of stock solution (6.2) to 1 liter in a volumetric flask.
6.4 Boric acid solution (20 g/L): Dissolve 20g H_3BO_3 in distilled water and dilute to 1 liter.
6.5 Nessler reagent: Dissolve 100 g of mercuric iodide and 70 g of potassium iodide in a small amount of water. Add this mixture slowly, with stirring, to a cooled solution of 160g of NaOH in 500 mL of water. Dilute the mixture to 1 liter. If this reagent is stored in a Pyrex bottle out of direct sunlight, it will remain stable for a period of up to 1 year. *NOTE: This regent should give the characteristic color with ammonia within 10 minutes after addition, and should not produce a precipitate with small amounts of ammonia (0.04 mg in a 50 mL volume).*
6.6 Borate buffer: Add 88 mL of 0.1 N NaOH solution to 500 mL of 0.025M sodium tetraborate solution (5.0 g anhydrous Na2B4O7 or 9.5g $Na_2B_4O_7 \bullet 10H_2O$ per liter.) and dilute to 1 liter.
6.7 Sulfuric acid, standard solution: (0.02 N, 1mL = 0.28 mg NH3-N). Prepare a stock solution of approximately 0.1 N acid by diluting 3 mL of conc. H_2SO_4 to 1 liter with CO_2-free distilled water. Dilute 200 mL of this solution to 1 Liter with CO_2 free distilled water. *NOTE: An alternate and perhaps preferable method is to*

standardize the approximately 0.1 N H_2SO_4 against a 0.100 N $NaCO_3$ solution. By proper dilution the 0.02 N acid can then be prepared.

6.8 Sodium hydroxide Solutions

6.8.1 1N solution: Dissolve 40 g NaOH in ammonia-free water and dilute to 1 Liter.

6.8.2 10N solution: Prepare 10N solution by dissolving 40-g in 100-mL.

6.9 Dechlorinating reagents: A number of dechlorinating reagents may be used to remove residual chlorine prior to distillation. These include:

a. Sodium thiosulfate (1/70 N): Dissolve 3.5g $Na_2S_2O_3 \bullet 5H_2O$ in distilled water and dilute to 1 Liter. One mL of this solution will remove 1 mg/L of residual chlorine in 500 mL of sample.

b. Sodium arsenite (1/70 N): Dissolve 1.0g $NaAsO_2$ in distilled water and dilute to 1 liter.

7.0 PROCEDURE

7.1 Distillation

7.1.1 Preparation of equipment: Add 50 mL of distilled water to an 80 mL Kjeldahl flask. The addition of boiling chips, which have been previously treated with dilute NaOH, will prevent bumping. Steam out the distillation apparatus until the distillate shows no trace of ammonia.

7.1.2 Sample preparation: Remove the residual chlorine in the sample by adding dechlorinating agent equivalent to the chlorine residual. To 40 mL of sample add 1N NaOH until the pH is 9.5, checking the pH during addition with a pH meter or by use of a short-range pH paper.

7.1.3 Transfer the sample, the pH of which has been adjusted to 9.5, to an 80 mL Kjeldahl flask and add 25 mL of the borate buffer. Distill 30 mL at the rate of 6-10 mL/min into 5 mL of 2% boric acid contained in a 125 mL Erlenmeyer flask. *NOTE: The condenser tip or an extension of the condenser tip must extend below the level of the boric acid solution.* Dilute the distillate to 50 mL with distilled water and nesslerize an aliquot to obtain an approximate volume of the ammonia-nitrogen concentration.

7.2 Standards Preparation - Prepare a series of standards as shown in the following table:

mL of Standard 1.0 mL = 0.01 mg NH_3-N	mg NH3-N/50-mL
0.0	0.0
0.5	0.05
1.0	0.10
2.0	0.20
3.0	0.30
4.0	0.40
5.0	0.50
8.0	0.80
10.0	1.0
20.0	2.0
50.0	5.0

7.3 Calibration of the Electrometer

7.3.1 Place 50-mL of each standard solution in a clean 100-mL beakers. Immerse electrode into standard of lowest concentration and add 0.5-mL of 10N sodium hydroxide solution while mixing. Keep electrode in solution until a stable reading is obtained.

7.3.2 **NOTE-1:** The pH of the solution after the addition of 10N NaOH must be above 11. Adjust the amount of 10N NaOH solution upwards (to 1-mL), if necessary. Use the same amount for samples and standards.

7.3.3 **NOTE-2:** Since ammonia may be lost from a basic solution, NaOH should not be added prior to immersing the electrode in the sample.

7.3.4 Obtain reading for each standard, going from the lowest to the highest. Plot log of the ammonia concentration against (use an Excel spreadsheet) against the electrode potential. Perform linear regression to obtain slope and intercept. The correlation coefficient should be greater than 0.995.

7.4 Sample Measurement

7.4.1 Follow the procedure described in section 7.3.1 exactly and record the stabilized potential for each sample.

7.4.2 Calculate sample ammonia concentrations from the curve (Section 7.3.4).

7.4.3 Report sample amounts (mg/L) as NH_3-N.

8.0 **REFERENCES**

8.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Nitrogen, Ammonia, Method 350.2 (Colorimetric, Titrimetric, Potentiometric), Distillation Procedure, Issued, 1971, and Editorial Revision 1974.
8.2 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Nitrogen, Ammonia, Method 350.3 (Potentiometric, Ion Selective Electrode), Issued, 1974.
8.3 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Method 4500-NH_3-A, 4500-NH_3-B, 4500-NH_3-E and 4500-NH_3-F.
8.4 AML Standard Operating Procedures, I-Ammonia-RB.
8.5 AML Standard Operating Procedures, I-TKN-RB.
8.6 AML Standard Operating Procedures, Q-Water-RC.
8.7 AML Standard Operating Procedures, Q-WateMgmt-RD.
8.8 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
8.9 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-Ammonia-350.2-RC

Ammonia by Distillation and Colorimetry (EPA 350.2)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

NITROGEN, AMMONIA (EPA Method 350.2)

1.0 SCOPE AND APPLICATION

1.1 This SOP is based on the distillation and colorimetric method based on EPA 350.2 and Standard Method 4500. This SOP covers the determination of ammonia-nitrogen exclusive of total Kjeldahl nitrogen, in drinking, surface and saline waters, domestic and industrial wastes.

1.2 It is the method of choice where economics and sample load do not warrant the use of automated equipment.

1.3 The method covers the range from about 0.05 to 1.0-mg NH_3-N/L for the colorimetric procedure.

2.0 SUMMARY OF METHOD

2.1 The sample is buffered at a pH of 9.5 with a borate buffer in order to decrease hydrolysis of cyanates and organic nitrogen compounds, and is then distilled into a solution of boric acid. The ammonia in the distillate can be determined colorimetrically by nesslerization.

3.0 SAMPLE HANDLING AND PRESERVATION

3.1 Samples may be preserved with 2-mL of conc. H_2SO_4 per liter and stored at 4 ^oC.

4.0 INTERFERENCES

4.1 A number of aromatic and aliphatic amines, as well as other compounds, both organic and inorganic, will cause turbidity upon the addition of Nessler reagent, so direct nesslerization (i.e., without distillation), has been discarded as an official method.

4.2 Cyanate, which may be encountered in certain industrial effluents, will hydrolyze to some extent even at the pH of 9.5 at which distillation is carried out. Volatile alkaline compounds, such as certain ketones, aldehydes and alcohols may cause an off-color upon nesslerization in the distillation method. Some of these, such as formaldehyde, may be eliminated by boiling off at a low pH (approximately 2 to 3) prior to distillation and nesslerizatoin.

4.3 Residual chlorine must also be removed by pretreatment of the sample with sodium thiosulfate before distillation.

5.0 APPARATUS

5.1 An all-glass distilling apparatus with an 80-100 mL flask.
5.2 Spectrophotometer for use at 425-nm and providing a light path of 1 cm or more.
5.3 Nessler tubes: Matched Nessler tubes about 300 mm long, 17 mm inside diameter, and marked at 225 mm ± 1.5mm inside measurement from bottom.
5.4 Erlenmeyer flasks: The distillate is collected in 125-mL flasks. These flasks should be marked at the 25 and 50-mL volumes. With such markings, it is not necessary to transfer the distillate to volumetric flasks.

6.0 REAGENTS AND CHEMICALS

6.1 Distilled water should be free of ammonia. Such water is best prepared by passage through an ion exchange column containing a strongly acidic cation exchange resin mixed with a strongly basic anion exchange resin. Regeneration of the column should be carried out according to the manufacturer's instructions. NOTE: All solutions should be made with ammonia-free water.
6.2 Ammonium chloride, stock solution: 1.0mL = 1.0 mg NH_3-N. Dissolve 3.819 g NH_4Cl in distilled water and bring to volume in a 1-Liter volumetric flask.
6.3 Ammonium chloride, standard solution: 1.0 mL - 0.01mg. Dilute 10.0 mL of stock solution (6.2) to 1 liter in a volumetric flask.
6.4 Boric acid solution (20 g/L): Dissolve 20g H_3BO_3 in distilled water and dilute to 1 liter.
6.5 Nessler reagent: Dissolve 100 g of mercuric iodide and 70 g of potassium iodide in a small amount of water. Add this mixture slowly, with stirring, to a cooled solution of 160g of NaOH in 500 mL of water. Dilute the mixture to 1 liter. If this reagent is stored in a Pyrex bottle out of direct sunlight, it will remain stable for a period of up to 1 year. *NOTE: This regent should give the characteristic color with ammonia within 10 minutes after addition, and should not produce a precipitate with small amounts of ammonia (0.04 mg in a 50 mL volume).*
6.6 Borate buffer: Add 88 mL of 0.1 N NaOH solution to 500 mL of 0.025M sodium tetraborate solution (5.0 g anhydrous Na2B4O7 or 9.5g $Na_2B_4O_7 \bullet 10H_2O$ per liter.) and dilute to 1 liter.
6.7 Sulfuric acid, standard solution: (0.02 N, 1mL = 0.28 mg NH3-N). Prepare a stock solution of approximately 0.1 N acid by diluting 3 mL of conc. H_2SO_4 to 1 liter with CO_2-free distilled water. Dilute 200 mL of this solution to 1 Liter with CO_2 free distilled water. *NOTE: An alternate and perhaps preferable method is to standardize the approximately 0.1 N H_2SO_4 against a 0.100 N $NaCO_3$ solution. By proper dilution the 0.02 N acid can then be prepared.*
6.8 Sodium hydroxide, 1N: Dissolve 40 g NaOH in ammonia-free water and dilute to 1 Liter.
6.9 Dechlorinating reagents: A number of dechlorinating reagents may be used to remove residual chlorine prior to distillation. These include:

a. Sodium thiosulfate (1/70 N): Dissolve 3.5g $Na_2S_2O_3 \bullet 5H_2O$ in distilled water and dilute to 1 Liter. One mL of this solution will remove 1 mg/L of residual chlorine in 500 mL of sample.
b. Sodium arsenite (1/70 N): Dissolve 1.0g $NaAsO_2$ in distilled water and dilute to 1 liter.

7.0 PROCEDURE

7.1 Preparation of equipment: Add 50 mL of distilled water to an 80 mL Kjeldahl flask. The addition of boiling chips, which have been previously treated with dilute NaOH, will prevent bumping. Steam out the distillation apparatus until the distillate shows no trace of ammonia with Nessler reagent.

7.2 Sample preparation: Remove the residual chlorine in the sample by adding dechlorinating agent equivalent to the chlorine residual. To 40 mL of sample add 1N NaOH until the pH is 9.5, checking the pH during addition with a pH meter or by use of a short-range pH paper.

7.3 Distillation: Transfer the sample, the pH of which has been adjusted to 9.5, to an 80 mL Kjeldahl flask and add 25 mL of the borate buffer. Distill 30 mL at the rate of 6-10 mL/min into 5 mL of 2% boric acid contained in a 125 mL Erlenmeyer flask. *NOTE: The condenser tip or an extension of the condenser tip must extend below the level of the boric acid solution.* Dilute the distillate to 50 mL with distilled water and nesslerize an aliquot to obtain an approximate volume of the ammonia-nitrogen concentration.

7.4 Calorimetric Determination of ammonia in distillate: Prepare a series of Nessler tube standards as follows:

mL of Standard 1.0 mL = 0.01 mg NH_3-N	mg NH3-N/5.0 mL
0.0	0.0
0.05	0.005
0.10	0.010
0.20	0.020
0.30	0.030
0.40	0.040
0.50	0.050
0.80	0.080
1.0	0.100

Dilute each tube to 5 mL with distilled water, add 0.2 mL of Nessler reagent and mix. After 20 minutes read the absorbance at 425-nm against the blank. From the values obtained plot absorbance vs. mg NH3-N for the standard curve. Determine the ammonia in the distillate by nesslerizing 5 mL or an aliquot diluted to 5 mL and reading the absorbance at 425-nm as described above for the standards. Ammonia-nitrogen content is read from the standard curve.

8.0 CALCULATIONS

8.1 Spectrophotometric

$$\text{mg/L NH3-N} = \frac{A * 1000}{5} * \text{Dilution factor}$$

where:

A = mg NH3-N read from the standard curve

9.0 References

9.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Ammonia by Colorimetry, Method 350.2.
9.2 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998.
9.3 AML Standard Operating Procedures, Q-Water-RC.
9.4 AML Standard Operating Procedures, Q-WateMgmt-RD.

9.5 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
9.6 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-BOD-RC

BIOLOGICAL OXYGEN DEMAND

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

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Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan Date
QA Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This Analytical Management Laboratories (AML) standard operating procedure (SOP) for the determination of biological oxygen demand (BOD) is based on EPA Method 405.1 and Standard Method 5210B.

1.2 This SOP is applicable for determining the relative oxygen requirements of municipal and industrial wastewaters. Application of the test to organic waste discharges allows calculation of the effect of the discharges on the oxygen resources of the receiving water. Data from BOD tests are used for the development of engineering criteria for the design of wastewater treatment plants.

1.3 The BOD test is an empirical bioassay-type procedure, which measures the dissolved oxygen (DO) consumed by microbial life while assimilating and oxidizing the organic matter present. The standard test conditions include dark incubation at 20°C for a specified time period (5 days).

1.4 The actual environmental conditions such as temperature, biological population, water movement, sunlight, and oxygen concentrations cannot be accurately reproduced in the laboratory. Results obtained must take into account the above factors when relating BOD results to stream oxygen demands.

2.0 SUMMARY OF METHOD

2.1 The sample of wastewater, or an appropriate dilution, is incubated for 5 days at 20°C in the dark. The reduction in dissolved oxygen concentration during the incubation period yields a measure of the biochemical oxygen demand.

3.0 INTERFERENCES

3.1 A number of factors, for example, soluble versus particulate organics, settleable and floatable solids, oxidation of reduced iron and sulfur compounds, oxidation of reduced forms of nitrogen or lacking of mixing may affect the accuracy and precision of BOD measurements.

3.2 Nitrogenous demand historically has been an interference in the determination of BOD. If an inhibiting chemical is used, the oxygen demand measured is the carbonaceous demand (CBOD). If an inhibiting chemical is not used, the oxygen demand measured is the sum of the carbonaceous and nitrogenous demand, which is commonly reported as BOD_5.

4.0 APPARATUS AND MATERIALS

4.1 BOD bottle (300-mL) with glass stopper and plastic cap. Clean bottles with a detergent, rinse thoroughly, and drain before use.

4.2 Precision Scientific low temperature incubator Model 815, capable of maintaining 20°C. The incubator should exclude all light to prevent photosynthetic production of DO.
4.3 Dissolved Oxygen Meter (YSI Scientific Model 815), equipped with YSI Model 5905 BOD probe.
4.4 Magnetic stirring bars
4.5 Heat/Stir Plate, Fisher Scientific
4.6 Thermometer
4.7 Balance - Analytical, 0.0001 grams and top loading, 0.1 grams.
4.8 Disposable pipettes - Pasteur
4.9 Transfer pipettes, various volumes (1-mL, 2-mL, 5-mL, 10-mL, etc.)
4.10 Eppendorf pipettes, variable volume (100 to 1000-µL)
4.11 Parafilm
4.12 Five-gallon jug (Culligan) with 3-L graduation marks. Wash it with a small amount of bleach and rinse thoroughly. Clean it thoroughly with a special detergent formulated for cleaning BOD bottles, rinse it thoroughly with tap water and deionized water. Keep it free of microorganisms.
4.13 Air pump and airstone, to aerate dilution water. Wash the airstone periodically as described in Section 4.12.

5.0 REAGENTS

5.1 It is assumed that all inorganic/organic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where available. Any other reagents must demonstrate high purity without lessening the accuracy of the determination.
5.2 Reagent water - All references to water in this method will be assumed to be ASTM Type I or Type II reagent water, except when referring to field samples. See AML SOP on reagent water
5.3 Dilution Water - This is prepared using commercially available nutrients and Type II water.
5.3.1 Nutrient buffer pillow (Hach Company, catalogue # 14861-98). The pillows contain granules of the common nutrient chemicals (ammonium chloride, calcium chloride, ferric chloride, magnesium sulfate, sodium and potassium phosphates, mono- and di-basic) in a pouch.
5.3.2 To three liters of Type II DI water (source water) at 20 ± 3°C in a 5-gallon jug, add one buffer pillow and aerate with airstone (Section 4.13). Continue to aerate the dilution water during the entire time it is to be in use. Keep lights out to prevent algae growth.
5.3.3 Prepare new dilution water every six hours. Discard dilution water that is more than 24-hours old.
5.3.4 Replace source water if the dilution water blank shows more than 0.2-mg/L DO depletion in 5 days. Clean the BOD bottles (and 5-gallon jug used to store dilution water as described earlier) with specially formulated detergent and <u>rinse thoroughly</u> before starting a new batch of sample analysis.

5.4 Seed Water - This is prepared using commercially available seed and Type II water as described below.

5.4.1 Seed (Hach Company, catalogue #2471200). The seed contains microorganisms capable of oxidizing biodegradable organic matter in a capsule form.

5.4.2 Add one capsule seed to 1-L dilution water (Section 5.3.2). Stir for one hour before use. Use portions of the capsule for smaller amounts of dilution water. NOTE: If desired, the wastewater treatment plant can provide seed for analysis of their wastewater stream.

5.4.3 Prepare new seed water every six hours.

5.5 GGA Standard (300 mg/L) - This is prepared by dissolving 150-mg glucose and 150-mg glutamic acid in 1-L Type II water. Store at 4°C when not in use. Replace the standard solution after 30-days.

5.5 Blank - This is prepared by filling a clean 300-mL BOD bottle with freshly prepared dilution water (Section 5.3).

5.6 Blank with Seed - This is prepared by placing 2-mL of seed water (Section 5.4) in a clean 300-mL BOD bottle and filling it with freshly prepared dilution water (Section 5.3). Prepare blank with seed in triplicate.

5.7 Laboratory Control Samples (LCS) - This is prepared by placing 6-mL of the GGA standard solution (Section 5.5) and 2-mL of the seed water (Section 5.4) in a clean 300-mL BOD bottle and filling it with freshly prepared dilution water (Section 5.3). The concentration of glucose and glutamic acid should be 3-mg/L each in the LCS. Prepare LCS in triplicate.

5.6 Matrix Spike Samples (MS and MSD) - Matrix spike analyses are not commonly performed with this analysis. If required, these can be prepared by substituting the GGA standard with an appropriate amount of sample. The analyst discretion should be used in selecting the volume of sample used. Prepare MS in duplicate.

5.7 Sodium hydroxide - Prepare 1N solution by dissolving 40-g in reagent water and diluting it to 1 L.

5.8 Sulfuric acid - Dilute 28-mL in reagent water (1 L) with stirring.

5.9 Sodium sulfite - Dissolve 0.1575-g Na_2SO_3 in 100-mL of reagent water. Prepare fresh daily since the solution is not stable.

5.10 Starch-potassium iodide paper (Fisher scientific).

5.11 TCMP - Purchase 2-chloro-6-(trichloromethyl) pyridine (TCMP) from Aldrich Chemicals.

5.12 Glucose, reagent grade.

5.13 Glutamic acid, reagent grade.

5.14 Detergent for cleaning BOD bottles.

6.0 SAMPLE PRESERVATION HANDLING & STORAGE

6.1 One liter plastic bottles with polyethylene screw caps may be used for collecting water samples. Order Level-6 pre-cleaned 32-Oz round w/m HDPE containers (QEC catalogue #6213-003). Provide 1 bottle for each water sample. Quality control samples such as MS and MSD are not usually collected for this test.

6.2 Upon receipt and log-in, these sample containers should be stored in the walk-in cooler at 4°C. The analyst should be alerted immediately due to short holding times.
6.3 Sample analysis must begin within 48-hours of sample collection, preferably as soon as samples are received.

7.0 HEALTH AND SAFETY

7.1 Samples may contain high levels of volatile and/or semivolatile organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.
7.2 Sample bottles, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.
7.3 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.
8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.
8.3 Unused aqueous samples can be disposed off into the laboratory sink.

9.0 **PROCEDURE**

The procedure involves checking the dilution water, controlling the seed amount, sample pretreatment (when necessary), sample dilution and dissolved oxygen (DO) determination on day1 and day 5 after incubation at 20°C.

9.1 Dilution water control - The 5-day DO uptake should not exceed 0.2-mg/L. Type I water may be used if Type II water is unsuitable to prepare dilution water. The DO meter calibration should be checked and re-calibrated if necessary while measuring small changes in the DO readings.
9.2 Seed control - The DO uptake attributable to the seed added to each bottle should be between 0.6 and 1.0-mg/L. Prepare LCS samples as described in Section 5.7 and dose them with various volumes (e.g. 0.5-mL, 1.0-mL, 2.0-mL, 5.0-mL) of seed water (Section 5.4). From a plot of the depleted DO versus the seed water volume, calculate the DO depleted per mL of the seed water. The actual volume of seed water added to all the BOD bottles should be adjusted to provide GGA check results (LCS samples) in the 198 ± 30-mg/L range. The default seed water volume to be used is 2-mL.
9.3 Sample pretreatment - Samples should warm up to the room temperature before analysis could begin. Perform the following checks.
    9.3.1 Check sample pH. Neutralize samples to pH 6.5 to 7.5 with dilute sulfuric acid or sodium hydroxide. Samples should not be diluted by more than 0.5% during pH adjustment.

9.3.2 Check samples for residual chlorine with starch-potassium iodide paper (Section 5.10). Treatment is not required if the residual chlorine level is less than 10-ppm. Excess chlorine should be neutralized with the right amount of sodium sulfite (Section 5.9). See AML SOP on percent chlorine to determine the correct amount to be added to the sample. The samples for BOD analyses should be collected before chlorine treatment.

9.3.3 Samples containing toxic substances (e.g. heavy metals) may need special study and treatment. Contact client for details.

9.3.4 Samples containing supersaturating levels of DO - Warm the samples to 20°C and shake partially filled bottles vigorously to aerate them.

9.3.5 Nitrification inhibition - This is performed only when carbonaceous biological oxygen demand (CBOD) results are required. Add 3-mg of TCMP (section 5.11) to each 300-mL BOD bottle (10-mg/L in the diluted sample) before capping. This step should be skipped for BOD measurements.

9.4 Prepare Blank as described previously (Section 5.5).

9.5 Prepare seed blank in triplicate as described previously (Section 5.6).

9.6 Prepare LCS in triplicate as described previously (Section 5.7).

9.7 Sample Dilution - Make several dilutions of sample that will result in a residual DO of at least 1-mg/L and a DO uptake of at least 2-mg/L after a 5-day incubation at 20°C. The number of dilutions needed depend on the specific sample.

9.7.1 The number of dilutions may be minimized by quickly measuring the COD (See AML SOP) and estimating the BOD at 60% of the COD result.

9.7.2 Use the following default dilutions in the absence of prior knowledge about the wastestreams: 0.0 to 1.0% for strong industrial wastes, 1 to 5% for raw and settled wastewater, 5 to 25% for biologically treated wastewater, and 25 to 100% for polluted river waters.

9.7.3 For samples that are analyzed routinely and repetitively (e.g. Front Street Plant weekly samples) analyze four dilutions (e.g. 50-mL, 100-mL, 200-mL, and 300-mL) with two of the dilutions in duplicate. The dilution scheme should be based on historical results for the specific sample.

9.7.4 For samples that are analyzed routinely and repetitively and are known to contain high levels of BOD (e.g. ESI tank Farm), use larger dilutions (e.g. 0.05-mL, 0.1-mL, 1.0-mL, and 5.0-mL).

9.7.5 For unknown samples, make at least six dilutions with two dilutions in duplicate. Most common dilutions are: 0.1-mL, 0.3-mL, 1.0-mL, 3.0-mL, 10-mL, and 30-mL). The high dilutions (0.1 and 0.3-mL) may be replaced by other sample volumes (100-mL and 300-mL) for samples not suspected to contain high levels of BOD.

9.7.6 Prepare dilutions directly in BOD bottles.

9.8 To each sample or the sample dilution (Section 9.7), add 2-mL (or other volumes as determined in Section 9.2) of seed water (Section 5.4). Fill each 300-mL BOD bottle with enough dilution water (Section 5.3) so that insertion of stopper will displace all air, leaving no bubbles. (NOTE: The insertion of membrane electrode may result in the displacement of the content. The volume of diluent should be adjusted to reduce

overflow.) The seed water and dilution water used in this procedure must be identical to those used for the preparation of blank, blank with seed and LCS.

9.9 Measure and record the DO of each sample and standard prepared as described in Section 9.4, 9.5, 9.6 and 9.8. Replace any displaced contents from the insertion of electrode with dilution water.

9.10 Place glass stopper on bottle making sure that there are no air bubbles. Place the plastic cap (Parafilm may be used if caps are unavailable) over the bottle. Put the bottles in the incubator at 20°C for 5 days. Monitor the incubator temperature every day. Measure and record the DO of each bottle after the 5th day.

10.0 CALCULATIONS

10.1 Calculate the DO depletion, B in each seeded blank by subtracting the DO result, B_2 on Day 5 (after incubation) from the DO result, B_1 on Day 1 (before incubation).

$$B = (B_1 - B_2)$$

10.2 Calculate the mean blank correction (B_m) from triplicate analysis of the seeded blank.

10.3 Calculate the DO depletion, D in each sample dilution by subtracting the DO result, D_2 on Day 5 (after incubation) from the DO result, D_1 on Day 1 (before incubation). Calculate D only for each test bottle that meet the 2.0-mg/L minimum depletion and the 10-mg/L residual DO (D_2>1.0) requirement.

$$D = (D_1 - D_2)$$

10.4 Calculate BOD_5 in mg/L using the following equation for each valid dilution of the sample, where V is the volume of the sample in mL and 300-mL is the default volume (no dilution):

$$BOD_5 \text{ (mg/L)} = (D - B_m) * (300/V)$$

10.5 When more than one sample dilution meets the criteria for DO depletion (>2-mg/L) and minimum DO (>1-mg/L) for measurement, the average result may be calculated and reported.

10.6 An Excel spreadsheet may be used to perform the calculations.

11.0 QUALITY CONTROL

Analytical data, which includes copies of the sample analysis logbook pages, computer printouts of the calculations, etc. should be gathered prior to review. Document steps such as the verification of calculations, deviations from the SOP procedures and other exceptions.

11.1 Summarize sample results using sample result standard EPA CLP Form 1.

11.2 The lowest possible detection limit based on the minimum DO depletion requirement (2-mg/L) is 2.0-mg/L. The laboratory MDL is 5-mg/L, which makes allowance for seeded blank results and the lowest result that is acceptable for DO measurement (1-mg/L).

11.3 The method quantitation limit is not applicable to this test. The laboratory should use five times the lowest possible detection limit (2-mg/L) as MQL. The laboratory MQL is 10-mg/L.

11.4 LCS results are not applicable for this test.

11.5 MS results are not applicable for this test.

12.0 **DATA VERIFICATION AND VALIDATION -** This section will provide guidance when evaluating and validating data generated by this SOP. It provides procedures to assess if data produced is valid and legally defensible.

12.1 HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection from the chain of custody to the time of sample preparation and/or analysis.

12.1.1 WATER - Aqueous samples are collected unpreserved. The holding time is 48 hours from sampling.

12.1.2 SOIL – This test is not applicable to soil samples.

12.1.3 ACTION – If holding times have been exceeded then all positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or invalid (I).

12.1.4 ACTION – If samples were not received by the laboratory within 48-hours of collection, inform the client about the holding time problem. Sample collection for BOD should be scheduled in the middle of the week (preferably on Wednesdays and Thursdays) so that Day 1 and Day 5 measurements may be performed by the laboratory during weekdays.

12.2 DILUTION WATER BLANKS – The assessment of dilution water blank analysis is to determine the degree of potential microbial and organic contamination introduced during the analysis.

12.2.1 Dilution blanks should have a Day 1 reading between 8 and 9-mg/L. Ideally, the DO depletion should not exceed 0.2-mg/L.

12.2.2 ACTION – Wash the 300-mL BOD bottles and containers used to store reagent water and/or the dilution water thoroughly to remove microorganisms and organic matter for future BOD analysis batches.

12.3 SEEDED DILUTION WATER BLANKS – The assessment of seeded dilution blank analysis is to determine the degree of potential organic contamination introduced during the analysis and to correct the DO depletion in samples.

12.3.1 Seeded dilution water blanks should have a Day 1 reading between 8 and 9-mg/L. Ideally, the DO depletion should not exceed 2 to 3-mg/L.

12.3.2 ACTION – Wash the 300-mL BOD bottles and containers used to store reagent water and/or the dilution water thoroughly to remove microorganisms and organic matter.

12.3.3 ACTION – Adjust the seed amount as described in Section 9.2 for future BOD analysis batches.

12.4 DILUTION SCHEME - In some unknown samples, it is possible that none of the dilutions performed may meet DO depletion and the residual DO requirements. Use the highest dilution for BOD calculation and 1-mg/L as the final DO when the 5-day DO readings are <1-mg/L. Report the calculated BOD result as >RESULT. For example, if the calculated result is 30,000-mg/L report it as >30,000-mg/L.

13.0 REFERENCES

13.1 Methods for the Chemical Analysis of Water and Wastes, US EPA, EPA-600/4-79-020, Method 405.1.

13.2 Standard Methods for the Examination of Water and Wastewater, 20th Edition, Method 5210B.

13.3 AML Standard Operating Procedures, Q-Water-RC.

13.4 AML Standard Operating Procedures, Q-WasteMgmt-RD.

STANDARD OPERATING PROCEDURES

No. I-Chloride-Titrimetry-RC

Chloride by Titrimetry
(EPA 325.3, SM 4500-CL$^-$-B, and SW-846 9077/9253)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

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Laboratory Director, Analytical Management Laboratories Date

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Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Chloride by Titrimetry
(SM 4500-CL$^-$-B, and SW-846 9077/9253)

1.0 Scope and Application

1.1 This SOP is based on Standard Methods 4500-Cl$^-$-B, SW-846 Method 9077, EPA 325.3, and SW-846 Method 9253 for the determination of chloride by titrimetry.
1.2 This method is applicable to drinking, surface and saline waters, domestic and industrial wastes.
1.3 Additional procedures included in this SOP based on SW846 Method 9077 would make the procedures applicable to new or used petroleum products for the determination of their total halogen measured as chloride. The SW846 procedure involves methods for converting all organic halogens to their respective sodium halides.
1.4 This SOP is also applicable to oxygen bomb combustates or other waters where the chloride content is 5 mg/L or more.
1.5 The titrimetric method is suitable for all concentration ranges of chloride content; however, in order to avoid large titration volume, oil sample aliquots containing not more than 4000 mg/kg should be used.
1.6 Sample aliquots (50-mL) for titration should preferably contain chloride at levels greater than 5-mg/L but less than 20-mg.

2.0 Summary of Method

2.1 Water adjusted to pH 8.3 is titrated with silver nitrate solution in the presence of potassium chromate indicator. The end point is indicated by persistence of the orange-silver chromate color.
2.2 The oil sample (2-g) is dispersed in a solvent and reacted with a mixture of metallic sodium catalyzed with naphthalene and diglyme at ambient temperature. This process converts all organic halogens to their respective sodium halides. All halides in the treated mixture including those present prior to the reaction, are then extracted into an aqueous buffer, which is then titrated with silver nitrate as described in Section 2.1.

3.0 Comments

3.1 Bromide, iodide, and sulfide are titrated along with the chloride. Orthophosphate and polyphosphate interfere if present in concentrations greater than 250 and 25 mg/L, respectively. Sulfite and objectionable color or turbidity must be eliminated. Compounds that precipitate at pH 8.3 (certain hydroxides) may cause error by occlusion.
3.2 Free water, present as a second phase in oil samples should be removed. This second phase can be analyzed separately for chloride content if desired.
3.3 Residual sodium carbonate from the bomb combustion may react with silver nitrate to produce the precipitate, silver carbonate. This competitive reaction may interfere

with the visual detection of the end point. To remove carbonate from the test solution, add small quantities of sulfuric acid followed by agitation.

4.0 Apparatus

4.1 Standard laboratory titrimetric equipment including a 1 ml or 5 ml microburst with 0.01 ml graduations.
4.2 Plastic reaction bottle (100-mL)
4.3 Analytical balance: capable of weighing to 0.0001 g.
4.4 Class A volumetric flask: 1 L.

5.0 Reagents

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
5.2 Reagent water. All references to water in this method refer to reagent water (see AML SOP on water.
5.3 *Standard:* Purchase a prepared, certified 1000 mg/L standard. -------- OR --------- *Standard Sodium Chloride, 0.025N*: Dissolve 1.4613 g ± 0.0002g sodium chloride (dried at 600 ° for 1 hour) in chloride-free water in a l-Liter volumetric flask and dilute to the mark. 1mL=886.6 µg Cl.
5.4 *Nitric acid*, HNO_3 solutions (3 + 997)
5.5 *Sodium Hydroxide* solution (0.25N NaOH). Dissolve 10-g of NaOH in reagent water and dilute to 1 L with reagent water.
5.6 *Hydrogen peroxide* (30%), H_2O_2
5.7 Phenolphthalein indicator solution (10 g/L).
5.8 Potassium chromate indicator solution. Dissolve 50 g of potassium chromate (K_2Cr0_4) in 100 mL of reagent water and add silver nitrate ($AgN0_3$) until a slightly red precipitate is produced. Allow the solution to stand, protected from light, for at least 24 hours after the addition of $AgN0_3$. Then filter the solution to remove the precipitate and dilute to 1 L with reagent water.
5.9 Silver nitrate solution, standard (0.025N). Crush approximately 5 g of silver nitrate ($AgN0_3$) crystals and dry to constant weight at 40°C. Dissolve 4.2473 $\pm$ 0.0002 g of the crushed, dried crystals in reagent water and dilute to 1 L with reagent water. Standardize against the standard NaCl solution, using the procedure given in Section 7.0.
5.10 *Metallic sodium*: VWR Scientific, catalog # JT9410-1. Cut lump metallic sodium into 100-mg cubes and store them in mineral oil.
5.11 *Solvent mixture*: Prepare a solvent mixture containing the following organic compounds and solvent:

Stoddard solvent (commercial grade)-200-ml
Toluene (reagent grade) – 65-ml
Butyl ether (VWR cat. #: EM-BX1945-6), 65-ml
Diglyme (VWR cat. #: EM-BX0770-1), 65-ml
Naphthalene (VWR cat. #: JT2718-1) 32-g
Demusifier (commercial grade): 18-g
Store this solvent mixture appropriately labeled in amber containers tightly capped to minimize oxidation. Use 8-ml of this solvent mixture for each sample.

5.12 *Phosphate Buffer*: Dissolve $NaH_2PO_4.2H_2O$ (46.3-g) in distilled water, add conc. HNO_3 (33.7-ml) and make it up to 1.0-l in a 1.0-L container. Use 50-ml of this buffer with each sample.

5.13 Sulfuric acid (1:19), H_2S0_4. Carefully add 1 volume of concentrated sulfuric acid to 19 volumes of reagent water, while mixing.

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6.0 Sample Collection, Preservation and Handling

6.1 All samples must have been collected using a sampling plan that addresses the data quality objectives of the project.

6.2 There are no special requirements for preservation

7.0 Procedure

7.1 Non-aqueous Samples

7.1.1 Weigh out 2-g of the non-aqueous sample (do not use the aqueous part, if the sample is biphasic) into a 100-ml reaction vessel. Add 8-ml of the solvent mixture (Section 5.11). Using a tweezer, add 500-mg (5x100-mg cubes) of metallic sodium (Section 5.10) into the reaction vessel. Samples containing water will react violently with sodium, generating heat and gas. Let the gases escape before putting on the cap. Shake the reaction vessel in a mechanical shaker for 5 minutes, allowing gases to escape as many times as necessary.

7.1.2 Add 50-ml of phosphate buffer (Section 5.12) to the reaction vessel. NOTE: Unreacted sodium will react with water releasing heat and gases. Release the gases, cap the reaction bottle and shake gently for a few seconds. Release the gases and shake for 30-seconds.

7.1.3 Use 10-mL of sample diluted to 50-mL with distilled water, so that the concentration of chloride does not exceed 20 mg aliquot.

7.2 Titration of Aqueous Samples and Aqueous Digestates

7.2.1 Pour 50 mL of sample or an aliquot of sample diluted to 50 mL with distilled water, containing between 0.25 mg and 20 mg of chloride ion, into a white porcelain container. Adjust the pH to the phenolphthalein end point (pH 8.3) using H_2S0_4 (Sec. 5.13) or NaOH solution (Sec. 5.5).

7.2.2 Add approximately 1.0 mL of K_2Cr0_4 indicator solution (Section 5.8) and mix. Add standard $AgN0_3$ solution (Section 5.9) drop-wise from a 25 mL burette until the orange color persists throughout the sample when

illuminated with a yellow light or viewed with yellow goggles. Be consistent with endpoint recognition.

7.2.3 Repeat the procedure described in Secs. 7.2.1 and 7.2.2 using exactly one-half as much original sample, diluted to 50 mL with halide-free water.

7.2.4 If sulfite ion is present, add 0.5 mL of H_20_2 (Section 5.6) to the samples described in Secs. 7.2.2 and 7.2.3 and mix for 1 minute. Adjust the pH, then proceed as described in Secs. 7.2.2 and 7.2.3.

8.0 Calculation

8.1 Calculate the chloride concentration as shown below:

$$\text{Chloride (mg/L)} = [(V_1 - V_2) \times N \times 71{,}000] / S$$

where:

V_1 = Milliliters of standard $AgN0_3$ solution added in titrating the sample prepared in Sec. 7.1.

V_2 = Milliliters of standard $AgN0_3$ solution added in titrating the sample prepared in Sec. 7.3.

N = Normality of standard $AgN0_3$ solution.

S = Milliliters of original sample in the 50 mL test sample prepared in Sec. 7.1.

71,000 = 2 x 35,500 mg Cl^-/equivalent, since $V_1 \sim 2V_2$.

8.2 Calculate the total chlorine concentration in the oil sample as shown below:

$$\text{Chloride mg/kg} = \frac{\text{(chloride, mg/L) * Volume of sample titrated (mL)}}{\text{Weight of the oil sample (g)}}$$

9.0 Quality Control

9.1 A Method Blank should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

9.2 A Second Source Standard (LCS) should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

9.3 A Sample Duplicate should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

10.0 References

10.1 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Chloride, Method 4500-Cl^--B.

10.2 Test Methods for Evaluating Solid Waste, Volume 1C: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9077, Test methods for Total Chlorine in New and Used Petroleum Products, September 1994.

10.3 Test Methods for Evaluating Solid Waste, Volume 1C: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9253, Chloride (titrimetric, Silver Nitrate), September 1994.

10.4 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.

10.5 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

10.6 AML Standard Operating Procedures, Q-Water-RC.

10.7 AML Standard Operating Procedures, Q-WasteMgmt-RD.

10.8 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

10.9 AML Standard Operating Procedures, Q-Controlcharts-RC.

10.10 AML Standard Operating Procedures, I-Anions-IC-RB.

10.11

STANDARD OPERATING PROCEDURES

No. I-Chlorine-330_4-RC

Total Residual Chlorine by Titrimtery (DPD-FAS) (EPA 330.4 and SM 4500-Cl)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

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Laboratory Director, Analytical Management Laboratories Date

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Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Total Residual Chlorine by Titrimtery (DPD-FAS) (EPA 330.4 and SM 4500-Cl)

1.0 Scope and Application

1.1 This SOP is based on EPA Method 330.4 and Standard Method 4500-Cl-F for the determination of total residual chlorine in water.
1.2 The N,N-diethyl-p-phenylene diamine (DPD) - ferrous ammonium sulfate (FAS) titration method is applicable to natural and treated waters at concentration above 0.1 mg/L Chlorine.

2.0 Summary of Method

2.1 Chlorine (hypochlorite ion, hpyochlorous acid) and chloramines stichiometrically liberate iodine from potassium iodide at pH 4 or less.
2.2 The iodine is titrated with FAS using DPD as the indicator.
2.3 The results are calculated as mg/L Cl even though the actual measurement is of total oxidizing power because chlorine is the dominant oxidizing agent present.

3.0 Interferences

3.1 Bromine, bromamine and iodine are interferences, which are normally present in insignificant amounts.
3.2 Oxidized manganese interferes but can be corrected by subtraction after performing a titration in the presence of sodium arsenite ($NaAsO_2$)
3.3 Copper interferes but is accounted for (up to approximately 10-mg/L copper) by incorporation of EDTA. The EDTA also retards deterioration of DPD due to oxidation and completely suppresses dissolved oxygen errors by preventing tract metal catalysis.
3.4 Turbidity and color may make the endpoint difficult to detect. Practice runs with spiked samples may be necessary.

4.0 Apparatus

4.1 Standard laboratory glassware is used. A microburet 0-2 mL or 0-10 mL is used depending on the desired range and accuracy.

5.0 Reagents

5.1 *Phosphate buffer solution:* Dissolve 2.4-g anhydrous disodium hydrogen phosphate, Na_2HPO_4, and 4.6-g anhydrous potassium dihydrogen phosphate, KH_2PO_4, in distilled water. Dissolve 0.080 g. Disodium ethylenediamine tetra-acetate dihydrate in 10 mL distilled water. Combine these two solutions and diluted to 100 mL with distilled water.

5.2 *N,N-Diethyl-p-phenylenediamine (DPD) indicator solution:* Dissolve 1 g DPD oxalate or 1.5 g p-amino-N,N-diethylaniline sulfate in chlorine free distilled water containing 8 mL of 1+3 H2SO4 and 200 mg disodium ethylenediamine tetra-acetate dihydrate. Dilute to 1 liter, store in a colored, glass-stoppered bottle. Discard when discolored. The buffer and indicator-sulfate are available as a combined reagent in stable powder form. CAUTION: The oxalate is toxic, avoid ingestion.

5.3 *Standard ferrous ammonium sulfate (FAS) titrant:* Dissolve 1.106g Mohr's salt $Fe(NH_4)_2(SO_4)_2 \bullet 6H_2O$ in distilled water containing 1 mL of 1+3 H_2SO_4 (5.4) and make up to 1 liter with freshly boiled and cooled distilled water. Stable for one month. Check with titration by standard potassium dichromate (5.5). The FAS titrant is equivalent to 100 ug Cl/1.00 mL.

5.4 *Sulfuric acid solution (1+3):* Slowly, add one part of H_2SO_4 to three parts of distilled water.

5.5 *Potassium dichromate (0.1000N):* Dissolve 0.4904g potassium dichromate ($K_2Cr_2O_7$) in distilled water and dilute to 100 mL.

5.6 *Potassium Iodide, KI Crystals.*

5.7 *Sodium Arsenite Solution:* Place 500 mg of sodium arsenite ($NaAsO_2$) in a flask and dilute to 100 mL with distilled water.

6.0 Procedure

6.1 This procedure gives a convenient direct reading (mL titrant = mg/L Cl) up to 4 mg/L. An aliquot should be diluted to 100 mL if higher concentrations are present.

6.2 Place 5 mL of phosphate buffer (5.1) in a titration flask.

6.3 Add 5 mL of DPD indicator (5.2)

6.4 Add approximately 1 g of KI (5.6) on a scoop.

6.5 Add 100 mL of sample.

6.6 Wait 2 minutes.

6.7 Titrate with FAS (5.3) until the red color is discharged. Record the volume of titrant used.

6.8 If oxidized manganese is present:

- 6.8.1 Place 5 mL of phosphate buffer (5.1) in a titration flask.
- 6.8.2 Add one small crystal of potassium iodide (5.6)
- 6.8.3 Add 0.5 mL of sodium arsenite solution (5.7)
- 6.8.4 Add 100 mL of sample. Mix.
- 6.8.5 Add 5 mL DPD indicator (5.2) Mix.
- 6.8.6 Titrate with FAS (5.3) until the red color is discharged. Record the volume of titrant used.

7.0 Calculation

7.1 The mL of FAS titrant is equal to the mg/L Cl. If oxidized manganese was present, subtract the amount of titrant used in 6.8.6 from the amount of titrant used in 6.7 to obtain the mg/L Cl.

8.0 Quality Control

8.1 A Method Blank should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.
8.2 A Second Source Standard (LCS) should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.
8.3 A Sample Duplicate should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

9.0 References

9.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Chlorine, Total residual, Method 330.4, Titrimetric, DPD-FAS, Issued, 1978.
9.2 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Method 4500-Cl-A and 4500-Cl-F.
9.3 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Method 4500-NH_3-A, 4500-NH_3-B, 4500-NH_3-E and 4500-NH_3-F.
9.4 AML Standard Operating Procedures, Q-Water-RC.
9.5 AML Standard Operating Procedures, Q-WateMgmt-RD.
9.6 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
9.7 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-Chromium-VI-RC

Hexavalent Chromium in Water and Alkaline Digestates by Manual Spectrophotometry (EPA 7196A, and SM 3500-Cr B)

November 2, 2005

APPROVED:

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Laboratory Manager, Analytical Management Laboratories Date

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Laboratory Director, Analytical Management Laboratories Date

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Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Hexavalent Chromium in Water and Alkaline Digestates by Manual Spectrophotometry (EPA 7196A, and SM 3500-Cr B)

1.0 Scope and Application

This SOP is used to measure hexavalent chromium (Cr-VI) present at high concentration levels in wastewater, groundwater, drinking water and TCLP leachates. Applicable concentration range is 0.05-mg/L to1.0-mg/L.

2.0 Summary of the Method

Cr-VI is reacted with an excess of 1,5-diphenylcarbazide (DPC) in acid solution to produce a red violet color. The absorbance at 540 nm is measured photometrically.

3.0 Sample Handling and Preservation

Store samples at 4°C. Holding time is 24 hours. Analyze samples immediately upon receipt.

4.0 Interferences

Hexavalent molybdenum and mercury at concentrations up to 200 mg/L; vanadium up to 10 times Cr-VI concentration.

5.0 Apparatus

Photometer with a greenish-yellow filter.
Test tubes (graduated to 10 ml) with screw caps

6.0 Reagents

Use only analytical reagent grade products unless otherwise specified.

DPC color reagent: Dissolve 250 mg of analytical reagent grade DPC in 50 ml of acetone in an amber bottle.

Sulfuric acid (10% v/v): Dilute 10-ml con. H_2SO_4 (acid) to 100 ml in reagent water.

Cr-VI Stock Standard: Dissolve 283 mg of dried potassium dichromate (K_2 Cr_2O_7) in 100 ml reagent water to prepare a primary stock, which is 1000 mg/L Cr-VI. Dilute 10 ml of the primary stock to 100 ml with reagent water to prepare the Matrix Spike Standard, which is 100 mg/L Cr-VI. Dilute 1ml of the primary stock to 100ml with reagent water to prepare a secondary stock, which is 10 mg/L Cr-VI. Use the secondary stock to prepare all the calibration standards.

Cr-VI Calibration Standards: These are prepared along with samples that require analysis for Cr-VI. The final volume of these standards and samples will be 10.00 ml before the addition of coloring reagent and acid.

(a) 0.00 mg/L STD (cal. blank) 0.00-ml secondary stock and 9.50-ml reagent water
(b) 0.05 mg/L STD - 0.05 ml secondary stock and 9.95 ml reagent water
(c) 0.10 mg/L STD - 0.10 ml secondary stock and 9.90 ml reagent water
(d) 0.25 mg/L STD - 0.25 ml secondary stock and 9.75 ml reagent water
(e) 0.50 mg/L STD - 0.50 ml secondary stock and 9.50 ml reagent water
(f) 0.75 mg/L STD - 0.75 ml secondary stock and 9.25 ml reagent water
(g) 1.00 mg/L STD - 1.00 ml secondary stock and 9.00 ml reagent water

7.0 Procedures

Use 13-mm x 150-mm disposable glass or clear plastic test tubes to perform color reaction and photometric measurements. Label the test tubes appropriately. Pipette 10.0-ml of the samples, LCS, method blank, spike sample or the calibration standards (as described above) into the test tubes. Measure and record absorbance (A_1) at 540-nm to determine the correction for sample turbidity. Add 0.20-ml of the DPC reagent. 0.05-ml to 0.30-ml acid to pH 2.0±0.5 (pH meter) and 0.25- to 0.00-ml reagent water. Let stand for 10 min. (stopwatch). Measure and record absorbance (A_2) at 540-nm again.

8.0 Calculations

Using an Excel spreadsheet, determine the slope (m), y-intercept (c) and correlation coefficient (R) for a plot of (A_2-A_1) in the y-axis against the Cr-VI concentration (0.00-mg/L to 1.0-mg/L) in the x-axis. Calculate sample concentration in mg/L using the following equation:

Sample Cr-VI concentration (mg/L) $= \{ (A_2-A_1) - c \} / m$

If Cr-VI concentrations exceed the upper calibration curve, dilute the sample appropriately with reagent water and reanalyze using the calibration curve generated as described above.

9.0 Quality Control

For each batch of 20 samples or less, perform the following quality control measurements:
1 method blank
1 laboratory control sample (LCS) at 0.50-mg/L Cr-VI from a second source
1 duplicate LCS
1 matrix spike (MS) fortified with Cr-VI at 0.50-mg/L (use 0.5-mL of MS standard)
1 MS duplicate

Control limits for LCS and MS recoveries are 85% to 115%. If LCS recoveries are outside the range, reanalyze LCS and associated samples, as applicable. If LCS recoveries are within the

control limits but MS recoveries are outside range, depending on the severity of deviation dilute samples either 5-fold (2.0-mL to 10.0-mL) or 10-fold (1.0-mL to 10.0-mL) with reagent water and repeat MS analysis. If the MS recoveries are still outside the range, check the pH of the raw sample, adjust it to a pH between 8.0 and 8.5 with NaOH and repeat MS analysis under dilution. If MS recoveries are outside 50% to 150%, contact the Operations Manager and the Project Manager to determine the appropriate course of action. Data may need to be reported with qualifications with suggestions to raise detection limits as appropriate. The applicability of alternate methods (e.g. SOP 7197.001) such as the chelation/extraction method may need to be explored. The latter is more sensitive and samples can be diluted to minimize the effect of interferences and reducing agents, which may be present in the sample.

10.0References

10.1Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Method 3500-Cr B.
10.2Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Alkaline Digestion for Hexavalent Chromium, Method 3060A, Revision 1, December 1996.
10.3Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chromium, Hexavalent, Colorimetric, Method 7196A, Revision 1, July 1992.
10.4AML Standard Operating Procedures, Q-Water-RC.
10.5AML Standard Operating Procedures, Q-WasteMgmt-RD.
10.6AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
10.7AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-COD-RC

Chemical Oxygen Demand by Manual Spectrophotometry (EPA 410.4, Hach 8000, and SM 5220)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

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Laboratory Director, Analytical Management Laboratories Date

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Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Chemical Oxygen Demand by Manual Spectrophotometry
(EPA 410.4, Hach 8000, and SM 5220)

1.0 Scope and Application

1.1 This SOP is based on EPA Method 410.4, Hach 8000 method and Standard Method 5220 for the determination of chemical oxygen demand (COD) by colorimetry in water samples. The procedures are based on EPA methods (410.4) as adapted by Hach Company, which provides simplified kits for this analysis.

1.2 This test measures the oxygen equivalents of the amount of organic matter in a waste sample, under specific conditions of oxidizing agent, temperature and time. The result has no definite relationship to the biochemical oxygen demand (BOD) or total organic carbon (TOC) levels.

1.3 The COD test results should be considered an independent measurement of organic matter in the sample, rather than as a substitute for the BOD or TOC results, which may be used for the development of engineering criteria for the design of wastewater treatment plants. This COD test can be applied to domestic and industrial wastewater containing COD in the 0 to 15,000-mg/L range.

2.0 Summary of Method

2.1 Organic and oxidizable inorganic substances in the sample are oxidized by potassium dichromate in 50% sulfuric acid solution in sealed tubes that are heated in an oven or block digester at 150°C. After two hours, the tubes are removed from the oven or digester, cooled and measured spectrophotometrically at 620-nm.

3.0 Interferences

3.1 Chlorides are quantitatively oxidized by dichromate and represent a positive interference. Mercuric sulfate is added to digestion tubes to complex the chlorides.

4.0 Sample Handling and Preservation

4.1 Collect samples (50-ml) in glass containers if possible. Use of plastic containers is permissible if it is known that no organic contaminants are present in the containers.

4.2 Samples should be preserved with sulfuric acid to pH <2 and maintained at 4 ^{o}C until analysis. The technical holding time is 28 days from the date of sample collection.

5.0 Apparatus

5.1 COD Reactor or a block digester
5.2 Safety Shield, laboratory bench
5.3 Test tube rack
5.4 Spectrophotometer, (Spectronic 20)
5.5 Muffle Furnace, 500°C

6.0 **Reagents**

6.1 Hach Digestion Solution for COD, 0 to 1500 mg/l range (cat. # 21259-15), package of 150 tubes from Hach company.
6.2 COD Standard Solution (1000 mg/l) from Hach (cat. # 22539-31)
6.3 CLS Solution (300 mg/l) from Hach Company (cat. # 12186-31)

7.0 **Procedure**

7.1 Shake the sample vial to homogenize and pipette 2.0-ml of the sample to the digestion solution (approx. 3-ml) contained in the Hach vial. NOTE: If the digestion solution turns green upon addition of the sample, a dilution (1:10) of the sample with distilled deionized water will need to be made prior to adding the sample to the digestion solution.
7.2 Prepare a method blank, calibration standards and LCS in the same manner as the samples. Cap the vials and invert them gently while holding the vials by the cap to mix the contents. NOTE: Perform this step over a sink since the vials may become very hot during mixing and improperly sealed vials may leak.
7.3 Heat the vials in a pre-heated block digester or a COD reactor at 150°C for 2 hours. Turn the reactor off, remove the vials, invert vials to mix their contents (while holding them by the cap) and allow them to cool in a test tube rack to room temperature.
7.4 Read the absorbance at 620-nm. For COD concentrations in the range 0 to 40 mg/l, read absorbance at 350-nm. A wavelength of 420-nm may be used for concentrations up to 150 mg/L.

8.0 **Calculations**

8.1 Prepare a standard curve by plotting absorbance at 620-nm (or other wavelengths as needed) against known concentrations of standards. Use an Excel spreadsheet to perform calculations and plotting of data.
8.2 The correlation coefficient for linear regression should be greater than 0.995. Calculate slope and intercept for linear regression.
8.3 Compute concentration of samples by comparing sample response to standard curve.

9.0 References

9.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Chemical Oxygen Demand, Method 410.4, Colorimetric, Automated/Manual, Issued 1978.
9.2 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Chemical Oxygen Demand, Method 5220-A and 5220-D.
9.3 Hach Company, Method 8000.
9.4 AML Standard Operating Procedures, Q-Water-RC.

9.5 AML Standard Operating Procedures, Q-WasteMgmt-RD.
9.6 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
9.7 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-Color-110_1-RC

Color by ADMI Filter Method (EPA 110.1 and SM 2120E)

November 2, 2005

APPROVED:

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Operations Manager, Analytical Management Laboratories Date

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Laboratory Director, Analytical Management Laboratories Date

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Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
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COLOR (METHOD 110.1, Colorimetric, ADMI)

1.0 Scope and Application

1.1 This SOP is based on EPA Method 110.1 and Standard Method 2120E for the determination of color using the American Dye Manufacturers Institute (AMDI) procedure.
1.2 This method is applicable to colored waters and wastes that have color characteristics significantly different from the yellow platinum-cobalt standard.
1.3 A working range of 25 to 250 color units is recommended. Sample values above 250 units may be determined by quantitative dilution.

2.0 Summary of Method

2.1 This method is an extension of the Tristimulus Filter Method. Tristimulus values are converted to an ADMI single number color difference, of the same magnitude assigned to plstnium-cobalt standards, using the Adams Nickerson Color Difference (DE).
2.2 Tristimulus values obtained by Spectrophotometric Method 204B may be used to calculate ADMI values as outlined in this procedure under Calculation 9.2.

3.0 Interferences

3.1 Since very slight amounts of turbidity interfere with the determination, turbid samples must be filtered prior to analysis. The optimum filter media to remove turbidity without removing color has not been found. Membrane and glass fiber filters with functional pore sizes of approximately 0.45 μ are convenient to use. Other techniques such as centrifuging and/or filtering aids may be used.

4.0 Sample Handling and Preservation

4.1 Since biological activity may change the color characteristic of a sample, the determination should be made as soon as possible. Refrigeration at 4 oC is recommended.

5.0 Calibration

5.1 Standard curves must be established (as outlined in Procedure 8.3) for each photometer used, and are not interchangeable. For color values less than 250, a 5 cm cell path is recommended. Less that 5 cm cell paths may be used if calibration is performed with the shorter cell.

6.0 Apparatus

6.1 Spectrophotometer or filter photometer capable of transmission measurements using tristimulus filters listed below:

Filter Number	Wavelength of maximum transmittance in nm	Corning designation*
1	590	CS 3-107
2	540	CS 4-98
3	438	CS 5-70

*Available from Corning Glass Works, Optical Products Department, Corning, NY 14830

7.0 Reagents

7.1 Standard chloroplatinate solution: Dissolve 1.246 g potassium chloroplatinate, K_2PtCl_6, (equivalent to 0.500 g metallic Pt) and 1 g crystalline cobaltous chloride, $CoCl_2 \bullet 6H_2O$, in distilled water containing 100 mL of conc. HCl. Dilute to 1000 mL with distilled water. This standard solution is defined as 500 ADMI color units.

7.2 Sulfuric acid, concentrated

7.3 Sodium hydroxide, 10 N: Dissolve 40 g of sodium hydroxide in 80 mL of distilled water. Cool to room temperature and dilute to 100 mL with distilled water.

8.0 Procedure

8.1 Prepare two 100 mL volumes of sample by maintaining the original pH of one aliquot and adjusting the second aliquot as necessary to pH 7.6 with sulfuric acid (7.2) or sodium hydroxide (7.3).

8.2 Filter samples to remove turbidity through 0.45 u membrane filter, glass fiber filter or other sutiable media (see interferences 3.1).

8.3 Use distilled water to set the transmittance at 100% and then determine the transmittance of the clarified sample or standard with each of the three tristimulus filters. Calibrate standards from 25 to 250 units are recommended.

9.0 Calculations

9.1 Calculate intermediate tristimulus values for samples and standards from the transmittance data in 8.3 using the following equations:

$X_s = (T_3 * 0.1899) + (T_1 * 0.791)$

$Y_s = T_2$

$Z_s = T_3 * 1.1835$

where:

T_1 = transmittance values in % using filter number 1
T_2 = transmittance values in % using filter number 2
T_3 = transmittance values in % using filter number 3

9.2 Convert tristimulus values to the corresponding Munsell values V_x, V_y and V_z by the use of published tables 2,3,4, or the equation suggested by Bridgeman.

9.3 Calculate DE values for samples and standards, construct a calibration curve by plotting DE against ADMI units of standards and determine ADMI color units of samples from the calibration curve.

$$DE = \sqrt{[0.23 * (V_{yc} - V_{ys})]^2 + [(V_{xc} - V_{xc}) - (V_{xs} - V_{ys})]^2 + [0.4[(V_{yc} - V_{sc}) - (V_{ysc} - V_{xs})]]^2}$$

where V_{xs}, V_{ys} and V_{zs} = the Munsell values for X_s, Y_s and Z_s respectively and V_{sc}, V_{yc} and V_{zc} = the Munsell values for a blank solution whose tristimulus values are X_c, Y_c and Z_c.

NOTE 1: If the photometer used is set at 100% transmittance with distilled water, the tristimulus values for the blank are 98.09, 100.00 and 118.35 for X_c, Y_c and Z_c respectively. If necessary, tristimulus values for the blank are determined as in calculation (9.1)

9.4 Report ADMI color values at pH 7.6 and at the original pH.

10.0References

10.1Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Color, Method 110.1, Issued 1978.
10.2Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Color, Method 2520E, ADMI Tristimulus Filter Method.
10.3AML Standard Operating Procedures, Q-Water-RC.
10.4AML Standard Operating Procedures, Q-WasteMgmt-RD.
10.5AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
10.6AML Standard Operating Procedures, Q-ControCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-Conductance-RC

Specific Conductivity
(EPA 120.1, SM 2510, and SW-846 9050A)

November 2, 2005

APPROVED:

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Operations Manager, Analytical Management Laboratories Date

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Laboratory Director, Analytical Management Laboratories Date

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Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Conductance (METHOD 120.1 μmhos at 25 ^{o}C)

1.0 Scope and Application

1.1 This SOP is based on EPA Method 120.1, Standard Method 2510, and SW-846 Method 9050A for the determination of specific conductance.
1.2 This method is applicable to drinking, surface and saline waters, domestic and industrial wastes and acid rain (atmospheric deposition).

2.0 Summary of Method

2.1 The specific conductance of a sample is measured by use of a self-contained conductivity meter.
2.2 Samples are preferable analyzed at 25 ^{o}C. If not, temperature corrections are made and result reported at 25 ^{o}C.

3.0 Sample Handling and Preservation

3.1 Analyses can be performed either in the field or laboratory.
3.2 If analysis is not completed within 24 hours of sample collection, sample should be filtered through a 0.45-micron filter and stored at 4 ^{o}C. Filter and apparatus must be washed with high quality distilled water and pre-rinsed with sample before use.

4.0 Comments

4.1 Instrument must be standardized with KCl solution before daily use.
4.2 Conductivity cell must e kept clean.
4.3 Field measurements with comparable instruments are reliable.
4.4 Temperature variations and corrections represent the largest source of potential error.

5.0 Apparatus

5.1 Conductivity bridge, range 1 to 1000 μmho per centimeter.
5.2 Conductivity cell, cell constant 1.0 or micro dipping type cell with 1.0 constant YSI #3403 or equivalent.
5.3 Thermometer

6.0 Reagents

6.1 Standard potassium chloride solutions, 0.01M: Dissolve 0.7456-g of pre-dried (2 hour at 105 ^{o}C) KCl in distilled water and dilute tot 1 liter at 25 ^{o}C.

7.0 Cell Calibration

7.2.1 The analyst should use the standard potassium chloride solution (6.1) and the table below to check the accuracy of the cell constant and conductivity bridge.

Conductivity 0.01 m KCl

o C	Micromhos/cm
21	1305
22	1332
23	1359
24	1386
25	1413
26	1441
27	1468
28	1496

8.0 Procedure

8.1 Follow the directions of the manufacturer for the operation of the instrument.
8.2 Allow samples to come to room temperature (23 - 27 ^{o}C), if possible.
8.3 Determine the temperature of samples within 0.5 ^{o}C. If the temperature of the samples is not 25 ^{o}C, make temperature correction in accordance with the instruction in section 9 to convert reading to 25 ^{o}C.

9.0 Calculations

9.1 These temperature corrections are based on the standard KCl solution.
9.1.1 If the temperature of the sample is below 25 ^{o}C, add 2% of the reading per degree.
9.1.2 If the temperature of the sample is above 25 ^{o}C, subtract 2% of the reading per degree.
9.2 Report results as Specific Conductance, µmhos/cm at 25 ^{o}C.

10.0References

10.1Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Conductance, Method 120.1, Editorial Revision, 1982.
10.2Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Conductivity, Method 2510A and 2510B.
10.3Test Methods for Evaluating Solid Waste, Volume 1C: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Specific Conductance, Method 9050A, December 1996.
10.4AML Standard Operating Procedures, Q-Water-RC.
10.5AML Standard Operating Procedures, Q-WateMgmt-RD.
10.6AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
10.7AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-Cyanide-RD

TOTAL CYANIDE AND CYANIDE AMENABLE TO CHLORINATION BY MANUAL DISTILLATION AND SPECTROPHOTOMETRY (EPA 335.1/335.2, SM 4500-CN^-, and SW-846 9010C/9014)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

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Quality Assurance Manager, Analytical Management Laboratories Date

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Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
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TOTAL CYANIDE AND CYANIDE AMENABLE TO CHLORINATION BY MANUAL DISTILLATION AND SPECTROPHOTOMETRY

(EPA 335.1/335.2, SM 4500-CN^-, and SW-846 9010B/9014)

1.0 Scope and Application

1.1 This Analytical Management Laboratories (AML) standard operating procedure (SOP) is based on EPA Methods 335.1 and 335.2, Standard Method 4500-CN^-, SW-846 Methods 9010C/9014, and EPA ILM 04.0, and ILM 05.0. The words, SOP, method and procedures are used interchangeably in this document. This method covers the determination of cyanide by manual distillation and spectrophotometric analysis of the distillate.

1.2 The procedure described in this SOP may be used as a reflux-distillation for both total cyanide and cyanide amenable to chlorination.

1.3 Prior to using this SOP, employees performing this preparation are encouraged to become familiar with underlying EPA methods to be sure they have a complete understanding of the method guidelines.

1.4 Cyanide determined by this method is defined as cyanide ion and complex cyanides converted to hydrocyanic acid by reaction in a reflux system with mineral acid in the presence of magnesium ion catalyst.

1.5 The detection limit for the colorimetric method is approximately 50-ug/L.

2.0 Summary of Procedure

2.1 The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined colorimetrically.

2.2 In the calorimetric measurement, the cyanide is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at pH less than 8 without hydrolysis to the cyanate. After the reaction is complete, color is formed upon the addition of pyridine barbituric acid reagent. The absorbance is read at 578-nm. To obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards.

2.3 Separate quality control procedures for the distillation part and the colorimetric analysis part are described in this SOP.

3.0 Interferences

3.1 All bottles must be thoroughly cleaned and rinsed to remove soluble materials from containers.

3.2 Oxidizing agents such as chlorine decompose most cyanides. Test a drop of the sample with potassium iodide-starch test paper (KI-Starch paper); a blue color indicates the need for treatment. Add ascorbic acid, a few crystals at a time, until a

drop of sample produces no color on the indicator paper. Then, add additional 0.6-g of ascorbic acid for each liter of sample volume.

3.3 Sulfide interference can be removed by adding an excess of bismuth nitrate before distillation. Treat samples that may potentially produce hydrogen sulfide with bismuth nitrate.

3.4 Nitrate and nitrite interference can be removed by pretreatment with sulfamic acid just before distillation.

3.5 Samples are preserved with 2-mL of 10 N sodium hydroxide per liter of sample (pH > 12) at the time of collection.

3.6 Samples must be stored at 4°C (± 2°C) and must be analyzed within the holding time.

4.0 Apparatus and Materials

4.1 Hot Block Digester (Environmental Express).

4.2 50-mL disposable digester cups (Environmental Express) with 10-ml, 25-ml and 50-ml calibration marks. Verify calibration as described in AML SOP on volumetric measurements.

4.3 Digester caps, disposable (Environmental Express)

4.4 Filters, disposable (Environmental Express)

4.5 Ribbed watch glasses for use with digester cups, disposable (Environmental Express)

4.6 pH meter

4.7 Vacuum source.

4.8 Refrigerator

4.9 Class A volumetric flasks, 100-mL, 250-mL, and 1000-mL

4.10 Erlenmeyer flask, 500-mL and 100-mL

5.0 Reagents and Standards

5.1 ASTM Type I water - See AML SOP on reagent water.

5.2 Sample Collection and Preservation Reagents

5.2.1 Sodium arsenite (0.1N), $NaAsO_2$. Dissolve 3.2-g of $NaAsO_2$ (Fisher cat #S225I-100) in 250-mL reagent water. Use it for removal of oxidizing agents.

5.2.2 Ascorbic acid (Fisher cat #A62-500).

5.2.3 Sodium Hydroxide (50%), Dissolve 50-g in reagent water and make up the volume to 100-mL in an Erlenmeyer flask.

5.3 Distillation and Preparation Reagents

5.3.1 Magnesium chloride solution, (51% w/v or 2.5M). Dissolve 510-g of $MgCl_2 \bullet 6H_2O$ (Fisher cat #M35-500) in ASTM Type II water and dilute to one liter.

5.3.2 Bismuth nitrate pentahydrate (Fisher cat #B337-500).

5.3.3 Glacial acetic acid (Fisher cat # A38-212), ACS Reagent Grade.

5.3.4 Bismuth Nitrate (3% w/v or 0.062M) Reagent. Dissolve 30-g of Bismuth Nitrate pentahydrate (Fisher) in 100-mL water, add 250-mL glacial acetic acid and dilute to 1-L with reagent water.

5.3.5 Sulfamic acid (18N), H_2NSO_3H Reagent. Dissolve 40-g of H_2NSO_3H (Fisher

cat #A295-500) in 1-L of reagent water.

5.3.6 Sulfuric acid (50% v/v or 18N). Carefully add a portion of concentrated H_2SO_4 to an equal portion of ASTM Type II water.

5.3.7 Sodium Hydroxide solution, 1.0N. Dissolve 40-g of NaOH in ASTM type I water and dilute to one liter.

5.3.8 Sodium Hydroxide solution, 0.025N. Dilute 25-mL of 1N NaOH in ASTM type I water and dilute to one liter.

5.3.9 Lead acetate test paper (Fisher cat #I4-862).

5.3.10 Nitrate/Nitrite Test Strips (Hach cat #27454-25).

5.4 Reagents for cyanides amenable to chlorination

5.4.1 Calcium hypochlorite (0.35M) solution - Dissolve 5-g of calcium hypochlorite (Fisher cat # C100-250), $Ca(OCl)_2$ in 100-mL of reagent water.

5.4.2 Sodium Hydroxide solution, 1.25N. Dissolve 50-g of NaOH in ASTM type I water and dilute to one liter.

5.4.3 Sodium arsenite (0.1N) - See Section 5.2.1

5.4.4 Potassium iodide starch paper (Fisher cat #14-860).

5.5 Reagents for Spectrophotometric Determination

5.5.1 Sodium Phosphate buffer solution, 1M.- Dissolve 138-g of $NaH_2PO_4 \bullet H_2O$ in ASTM Type I water and dilute to one Liter. Store at 4°C.

5.5.2 Chloramine-T solution - Dissolve 1.0-g of Chloramine-T in ASTM Type I water and dilute to 100-mL. Prepare fresh daily (at time of analysis).

5.5.3 Pyridine-barbituric acid Color Reagent Solution - Prepare this solution in the hood. Transfer 15-g of barbituric acid into a 250-mL volumetric flask. Add about 25-mL of ASTM Type I water and swirl the flask to mix. Add 75-mL of Pyridine and 15-mL concentrated HCl and mix until all the barbituric acid is dissolved. Dilute to 250-mL with ASTM Type I water and store at 4°C in the dark. This reagent should be replaced after six months of preparation.

5.5.4 Sodium Hydroxide solution, 0.25N. Dissolve 10-g of NaOH in ASTM type I water and dilute to one liter.

5.6 Standards

5.6.1 Document standard solution preparation and the preparation of reagents in the logbook (See Appendix 1 for details). Use the Calibration standard for LCS and MS analyses. Verify initial calibration curve (ICAL) with second source standards.

5.6.2 Standard Cyanide Calibration Solution, 1000 mg/L CN. Purchase from Solutions Plus (cat # 2985).

5.6.3 Intermediate Calibration Standard Solution (10 mg/L CN): Dilute 1-mL Standard Solution (5.6.2) with 99-mL 0.025N NaOH.

5.6.4 Laboratory Control Samples (LCS) - Two LCS samples (LCS-1 and LCS-2) are prepared at the low and high concentration levels of the initial calibration curve (Section 5.6.10). The LCS samples are distilled standards.

5.6.4.1 Low level LCS (LCS-1, 0.05-mg/L CN) - Dilute 0.5-mL of the 10-mg/L Intermediate Calibration Standard Solution (Section 5.6.3) to 100-mL with reagent water. Use 50-mL of this solution for the distilled LCS-1.

5.6.4.2 High level LCS (LCS-2, 0.20-mg/L CN) - Dilute 2.0-mL of the 10-mg/L Intermediate Calibration Standard Solution (Section 5.6.3) to 100-mL with reagent water. Use 50-mL of this solution for the distilled LCS-2.

5.6.5 Matrix Spike (MS) and MS Duplicate (MSD) Samples - Spike 50-mL of the sample selected for MS and MSD analyses with 0.5-mL of the 10-mg/L Standard (See Section 5.6.3). The default MS spike concentration of cyanide in the aqueous sample should be 0.1-mg/L.

5.6.6 Continuing calibration verification (CCV) Standard - The initial calibration curve is verified after every 10 samples using a mid-level un-distilled standard. Prepare CCV standard as described below:

5.6.6.1 Prepare 250-mL of CCV at 0.1-mg/L concentration by diluting 2.5-mL of the 10-mg/L Intermediate Calibration Standard Solution (Section 5.6.3) to 250-mL with reagent water. This mid level CCV standard is analyzed without distillation.

5.6.6.2 Use the CCV solution prepared as described in Section 5.6.6.1 for multiple analyses of the CCV, as needed.

5.6.7 Second source Stock Cyanide solution, 1000 mg/L CN. Dissolve 2.51g of KCN and 2.0g KOH in ASTM Type I water and dilute to one liter.

5.6.8 Second Source Intermediate Cyanide Solution, 10 mg/L CN. Dilute 1.0-mL of the Second source Stock Cyanide Solution plus 20-mL of 1.25N NaOH solution to 100-mL with ASTM Type I water.

5.6.9 Initial calibration verification (ICV) Standard - The initial calibration curve (See Section 5.6.10) is verified at the mid-level using a second-source un-distilled standard.

5.6.9.1 Prepare 50-mL of ICV at 0.1-mg/L concentration by diluting 0.50-mL of the Second-Source Intermediate solution (See Section 5.6.8) to 50-mL with reagent water. This mid level ICV standard is analyzed without distillation.

5.6.10 Initial Calibration (ICAL) Standards - Using the Intermediate Calibration Standard solution (5.6.3) prepare multi-point initial calibration (ICAL) standard solutions in 50-mL volumetric flasks as shown on the following Table. Prepare fresh daily. These are un-distilled standards.

Concentration of Cyanide in ICAL STD mg/L	Volume (mL) of 10-mg/L Standard Diluted to 50-mL
0.00	0.00
0.05	0.250
0.10	0.500
0.15	0.750
0.20	1.00
0.25	1.25
0.30	1.50

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Sample Preservation

6.1.1 Water Samples are preserved with 2-mL of 10 N sodium hydroxide per liter of sample (pH > 12) at the time of collection.

6.1.2 Soil samples are maintained at $4^{o}C$ ($\pm 2^{o}C$) until analysis.

6.2 Sample Containers. Order pre-cleaned bottles containing the 10N sodium hydroxide preservative directly from one of several vendors (e.g. cat #QEC 22120016NaOH). Provide one 16-Oz bottle for each water sample to be collected

6.2.1 Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples.

6.2.2 See project specific field sampling plan (FSP) for sample collection and preservation instructions.

6.3 Sample Preservation Check - Upon receipt and log-in, the tests for pH, oxidizing agents and sulfide should be performed.

6.3.1 The pH of the aqueous samples should be verified by the sample custodian and additional sodium hydroxide solution added as necessary to raise pH above 12.

6.3.2 The potassium iodide (KI)-Starch paper test for oxidizing agents should be performed by the analyst by testing a drop of the sample on the KI-starch paper. If the paper turns blue (iodide oxidized to iodine), add 0.1N sodium arsenite (Section 5.2.1) a few mL at a time until a drop of the sample produce no blue color on the indicator paper. Add an additional 1-mL of the arsenite solution for each 100-mL sample. Crystals of ascorbic acid (Section 5.2.2) may be used instead of the arsenite solution. NOTE: Sodium arsenite may have been added to the sample at the time of sample collection for some projects.

6.3.3 The lead acetate paper test for sulfide should be performed by the analyst by testing a drop of the sample on the test paper. If the paper turns black, add 5-mL of the 0.062M bismuth nitrate reagent (Section 5.3.4) to the distillation flask containing 50-mL sample. Do not treat the sample in the container.

6.3.4 The Nitrate/Nitrite water quality paper test strips should be used to determine their levels by the analyst by testing a drop of the sample on the test paper. If

the paper turns pink or if the samples are known to contain nitrate or nitrite (contact client project manager), add 5-mL of 0.4N sulfamic acid (Section 5.3.5) to the distillation flask containing 50-mL sample. Do not treat the sample in the container.

6.4 Holding Time is 14-days for soil and water samples from the date of sample collection.

6.4.1 Samples must be distilled and/or analyzed within the specified holding times for the results to be considered reflective of total concentrations.

6.4.2 Analytical data generated outside of the specified holding times must be considered to be minimum values only.

7.0 HEALTH AND SAFETY

7.1 Standards used for calibration or spiking may contain a number of toxic compounds at high concentrations. The toxicity of these complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Reagents used such as acids are highly corrosive. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.4 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.5 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Soil samples that have undergone extraction should be free of contaminants. They can be disposed off into sanitary dumpsters. Dispose of sample extracts containing high levels of acid in accordance with applicable AML SOPs.

9.0 PROCEDURE

Record measurements, observations, etc. on the sample preparation sheets contained in bound logbooks (see Appendix 6 for details). Record the lot numbers of all the reagents and standards used in sample preparation.

9.1 Sample Preparation - Retrieve samples to be analyzed from the cooler. Shake the sample thoroughly to homogenize any fine sediment within the sample container. Aqueous samples require the following checks prior to sample analysis:

9.1.1 Check for pH - See Section 6.3.1. If pH of the sample is unavailable (check with sample log-in data), determine pH using either a probe or paper and verify the sample has been properly preserved. If the sample is not preserved

at a pH >12, check with project manager for specific instructions.

9.1.2 Check for oxidizers - See Section 6.3.2 and treat sample as described.

9.1.3 Check for sulfide - See Section 6.3.3 and treat only the sample aliquot to be analyzed as described in the referenced section.

9.1.4 Check for nitrate/nitrite - See Section 6.3.4.

9.1.5 Check for the determination of cyanide amenable to chlorination - If this parameter is required two aliquots of the samples need to be analyzed. One sample should be distilled as described in Section 9.3 after subjecting the sample to the checks and adjustments described in Sections 9.1.1 to 9.1.5. Another aliquot should be pretreated as described in Section 9.2 prior to distilling the sample as described in Section 9.3. The cyanide amenable to chlorination is the difference between the observed results for the untreated and treated samples.

9.2 Pretreatment for cyanide amenable to chlorination

9.2.1 **NOTE:** Perform this operation in a hood since toxic gases (cyanogen chloride) are produced. Add calcium hypochlorite solution (See Section 5.4.1) dropwise with agitation to 50-mL sample, maintaining the pH between 11 and 12 with 1.25N NaOH (See Section 5.4.2) and testing for excess chlorine with KI-Starch paper (See Section 5.4.4). A distinct blue color should be maintained on the test paper for tests performed initially and after one hour of hypochlorite treatment. Additional hypochlorite may need to be added to maintain excess chlorine.

9.2.2 After 1-hour remove excess chlorine by adding 0.1N sodium arsenite (See Section 5.4.5) dropwise until KI-Starch paper shows no color.

9.2.3 The sample is now ready for distillation.

9.3 Distillation

9.3.1 The procedure described here uses a Midi distillation apparatus and requires a sample aliquot of 50-mL or less for aqueous samples and one gram for materials.

9.3.2 For aqueous samples: Pipette 50-mL of sample, or an aliquot diluted to 50-mL into the distillation flask along with 2 or 3 boiling chips.

9.3.3 For solid samples: Weigh 1.0-g of sample (±0.5-g) into the distillation flask and dilute to 50-mL with ASTM Type II water. Add 2 or 3 boiling chips.

9.3.4 At this time, all spike aliquots should be added to the appropriate QC samples. Spike each LCS, LCSD, MS and MSD sample with the spiking solutions.

9.3.5 Add 50-mL of 0.025N NaOH to the gas absorbing impinger.

9.3.6 Connect the boiling flask, condenser, and absorber in the train.

9.3.7 Turn on the vacuum and adjust the valves to give a flow of three-to-five bubbles per second from the impingers in each reaction vessel.

9.3.8 Treat samples with bismuth nitrate and/or sulfamic acid as described in Sections 9.1.2 to 9.1.4. See also Section 6.3.2 to 6.3.4.

9.3.9 After five minutes of vacuum flow, inject 5-mL of 50% H_2SO_4 through the top air inlet tube of the distillation head into the reaction vessel. Allow to mix for five minutes.

9.3.10 Add 2-mL of magnesium chloride solution through the top air inlet tube of

the distillation head into the reaction flask. Excessive foaming from samples containing surfactants may be quelled by the addition of another 2-mL of magnesium chloride solution.

9.3.11 After one and a half hours of refluxing, turn off the heat and continue the vacuum for an additional 15 minutes. The flasks should be cool at this time.

9.3.12 After cooling, close off the vacuum. Seal the receiving solutions and store them at 4°C until analyzed. The solution must be analyzed for cyanide within the 14-day holding time.

9.4 Color Development and Spectrophotometric Analysis

9.4.1 Transfer 25-mL of distillate or the standard (ICAL as described in Section 5.6.10, ICV1 and ICV2 as described in Section 5.6.8 and CCVs as described in Section 5.6.9) to a 50-mL volumetric flask.

9.4.2 Add 7.5-mL of 1 M NaH_2PO_4.

9.4.3 Add 1-mL of 0.4% (w/v) Chloramine-T solution and mix. Wait 2 minutes. NOTE: Some distillates may contain compounds that have chlorine demand. One minute after the addition of chloramine-T, test for excess chlorine with KI-starch paper. If the test is negative, add 0.25-mL chloramine-T. After one minute recheck with KI-starch paper. Continue to add chloramine-T in 0.25 mL increments until an excess is maintained.

9.4.4 After 1 to 2 minutes, add 2.5-mL Pyridine Barbituric Acid color reagent and mix well.

9.4.5 Dilute to 50-mL with ASTM Type I water. Wait 10 minutes, then read absorbance using spectrophotometer set at 578-nm.

9.4.6 Determine CN concentration using linear regression curve.

10.0 QUALITY CONTROL

10.1 The correlation coefficient for linear regression equation must be greater than or equal to 0.995 for the initial calibration (ICAL) to be acceptable.

10.2 Immediately after initial calibration with un-distilled standards, the calibration curve (ICAL) for the colorimetric analysis should be verified using an un-distilled, mid-level, second-source initial calibration verification (ICV) standard. The ICV results should be within ±15% of the true value (85% to 115% for %R) for ICAL to be acceptable.

10.3 Immediately after ICV analysis, the calibration curve (ICAL) should also be verified using an un-distilled, same-source, mid level continuing calibration verification (CCV) standard. The CCV results should be within ±10% of the true value (90% to 110% for %R).

10.4 Analyze an initial calibration blank (ICB) immediately after the analysis of the ICV. The analyte should not be present at levels greater than the lowest level for reporting (LLR). The LLR is one half of the method quantitation limit (MQL). The ICB will also serve as the MB for un-distilled samples containing only non-complexed cyanide.

10.5 Distilled samples containing cyanide in alkaline media and other samples containing only non-complexed cyanide may be analyzed with verified initial calibration curve.

10.6 Distilled samples should be accompanied by the following QC samples as described

below in detail: MB, LCS-1, LCS-2, MS, and MSD:

10.6.1 A method blank (MB) should be distilled with every batch of samples analyzed, or every 20 samples, whichever is greater.

10.6.2 Two same-source laboratory control samples (LCS-1 and LCS-2) representing the low and high end of the initial calibration curve should be distilled with every batch of distilled samples that are analyzed, or every 20 samples, whichever is less. See Section 5.6.4 for cyanide concentration that should be used in the low and high concentration LCS samples. The LCS results should be within ±10% of the true value (90% to 110% for %R) for the un-distilled standards. An aliquot of the un-distilled sample may be analyzed using the ICAL to obtain the true value of the LCS samples.

10.6.3 A matrix spike (MS) and matrix spike duplicate (MSD) should be distilled with every batch of distilled samples that are analyzed, or every 20 samples, whichever is greater. The MS recoveries should be within ±15% of the true value (85% to 115% for %R) for the spike.

10.6.4 A Sample Duplicate (MD) should accompany every batch of samples analyzed, or every 20 samples, whichever is greater. A MSD may be substituted for sample duplicates. The relative percent difference (RPD) for the duplicates should not exceed 20%.

10.7 Calculate sample concentrations from the standard curve. Report sample amounts greater than MDL (0.038-mg/L) or the reporting limit (0.05-mg/L) as appropriate. The MDL, MQLs, and QC limits for cyanide determination are shown in Tables 1 and 2.

11.0 REFERENCES

11.1 Methods for the Chemical Analysis of Water and Wastes, US EPA, EPA-600/4-79-020, Cyanide amenable to chlorination - titrimetric and spectrophotometric, Method 335.1.

11.2 Methods for the Chemical Analysis of Water and Wastes, US EPA, EPA-600/4-79-020, Cyanide, total - titrimetric and spectrophotometric, Method 335.2.

11.3 Methods for the Chemical Analysis of Water and Wastes, US EPA, EPA-600/4-79-020, cyanide, total - colorimetric, automated complexone, Method 335.3.

11.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9010B, Total and Amenable Cyanide, distillation, December 1996.

11.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9010C, Total and Amenable Cyanide, distillation, Revision 3, August 2002.

11.6 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9014, Titrimetric and Manual Spectrophotometric Determinative Methods for Cyanide, distillation, Revision 0, December 1996.

11.7 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 5, Miscellaneous methods, December 1996.

11.8 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 7, Characteristics Introduction and Regulatory Definitions, Chapter 7.3, Reactivity, December 1996.
11.9 EPA Contract Laboratory Program SOW ILM05.0, Exhibit D.
11.10 USEPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, ILM04.0, April 1994.
11.11 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.
11.12 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.
11.13 AML Standard Operating Procedures, Q-Water-RC.
11.14 AML Standard Operating Procedures, Q-WasteMgmt-RD.
11.15 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
11.16 AML Standard Operating Procedures, Q-Controlcharts-RC.

Table 1
List of Appendices

Appendix Number	Appendix Topic
1	Standards and Reagents Log
2	Initial Calibration (ICAL) Summary (Form-6 equivalents) Calibration Verification (ICV and CCV) Summary (Form-7 equivalents)
3	AML Checklist for Raw data Package Draft Case narrative Review Items, Criteria, and Optional Checklist for Data Verification
4	Method Detection Limit (MDL) Studies Summary
5	Sample Reports (Form-1 equivalents)
6	QC Association Forms (Form-4 equivalents) Sample Preparation Log Spectrophotometric Measurements Log
7	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
8	LCS-1/LCS-2 Sample Analysis Reports (Form-1 equivalents) LCS %R Summaries (Form-3 equivalents)
9	Method Quality Objectives for Method 9010/9012/9014 (AFCEE, USACE, DoD QSM, etc.)

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. I-DissolvedHydrogen-RC

Dissolved Hydrogen by Bubble Strip Method and Gas Chromatography

November 2, 2005

APPROVED:

_____ _____
Laboratory Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Dissolved Hydrogen by Bubble Strip Method

1.0 Scope and Application

1.1 This SOP is based on the research paper that us included in Appendix 1 for the determination of dissolved hydrogen using gas chromatography.

2.0 Summary of Method

2.1 The water sample for hydrogen analysis is collected using the bubble strip method and analyzed using gas chromatography as described in Appendix 1.

3.0 Interferences

See Appendix 1.

4.0 Apparatus

See Appendix 1.

5.0 Sample Collection, Preservation, and Handling

See Appendix 1.

6.0 Health, Safety, and Waste Disposal

6.1 Samples may contain high level of organic and inorganic contaminants. Gloves and safety glasses should be worn to minimize contact of the sample with skin and eyes.
6.2 Toxicity of the samples is not dependent on the oxygen content. Dispose of unused samples in accordance with AML waste disposal policies and procedures.

7.0 Calibration

See Appendix 1.

8.0 Procedure

See Appendix 1.

9.0 Calculations

See Appendix 1.

10.0References

10.1See Appendix 1.
10.2AML Standard Operating Procedures, Q-Water-RC.
10.3AML Standard Operating Procedures, Q-WasteMgmt-RC.

Appendix 1

(Article on dissolved hydrogen measurement)
(From the Proceedings of the 1998 Conference on Hazardous Waste Research)

DETERMINING DISSOLVED HYDROGEN, METHANE, AND VINYL CHLORIDE CONCENTRATIONS IN AQUEOUS SOLUTION ON A NANOMOLAR SCALE WITH THE BUBBLESTRIP METHOD

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1.0 SUMMARY

The Bubble Strip Method is a viable method for determining concentrations of hydrogen, methane, and vinyl chloride in aqueous solution. Information regarding the concentrations of these gases in groundwater is useful in monitoring bioremediation and predicting the fate of contaminants at a given site. Concentrations of dissolved gases on the nanomolar scale are measurable with this technique. Employing the method involves filling a gas sample bulb with the solution being analyzed, charging the bulb with a 20 mL headspace, and then pumping the solution through the bulb over a length of time sufficient forequilibrium between phases to be attained. Subsequent gas chromatographic analysis of the sample bulb headspace enables concentration of dissolved gas in solution to be calculated using Henry's law. The results of this study indicate that a solution flow rate of 400 mL/minute through the sample bulb is optimum. A flow time of twenty minutes is sufficient for equilibrium between phases to be established with aqueous solutions of hydrogen gas. With aqueous solutions of methane and vinyl chloride, equilibrium isattained within 10 minutes. A slightly longer time to equilibrium (about thirty minutes) was observed with solutions of hydrogen gas at 4°C.

2.0 INTRODUCTION

Intrinsic bioremediation of common groundwater contaminant fuels such as jet fuel, diesel fuel, and gasoline are a common source of groundwater contamination. At sites where spills have occurred, the slightly soluble organic fuel components are partitioned into the groundwater over potentially lengthy periods of time. The four contaminants which are of most concern are benzene, toluene, ethylbenzene, and xylenes (collectively known as BTEX). These compounds, while a relatively small percentage of the fuels by mass, account for most of the organic contaminants in fuel-tainted groundwater due to their high solubilities relative to other fuel components. The BTEX compounds pose a health risk and, therefore, are the primary focus of many cleanup efforts (Wiedemeier et al., 1996). A degreasing solvent, trichloroethylene (TCE), and the products of its degradation are also acommon source of contamination in groundwater. TCE can degrade to methane, ethane, andethylene with the hazardous compounds dichloroethylene and vinyl chloride as intermediates.

In some cases, natural processes are relied upon to aid in the remediation of groundwater contaminants. This strategy, known as intrinsic remediation, involves allowing natural attenuation processes to bring about a depletion of contaminant concentration over time. For an

example of asite where intrinsic bioremediation was found to be a viable option for restoration (see Kampbell et. Al.). Natural attenuation of contaminants occurs through a variety of physical, chemical, and biologi-cal processes. The processes of advection, dispersion, dilution from recharge, sorption, and volatilization do not change the identity of a contaminant, but serve to limit its potential as a biohazard by diluting it or transporting it to another location. A natural attenuation process which does change the identity of a contaminant, thereby rendering it harmless, is biodegradation. The process by which contaminants are biodegraded by microorganisms is referred to as intrinsic bioremediation. Hydrogen [H2] is an indication of the predominant pathway of biodegradation.

Intrinsic bioremediation occurs via mechanisms known as terminal electron accepting processes (TEAPs). TEAPs are microorganism mediated redox processes, the net result of which is the transfer of electrons from organic matter to a terminal electron acceptor. Terminal electron acceptors include O2 in aerobic conditions (i.e., [O2] > 0.5 mg/L), and CO2, NO3-, SO42-, Fe3+, and chlorinated hydrocarbons in anaerobic conditions. Molecular hydrogen is an intermediate in terminal electron accepting processes (Lovley, Chapelle, and Woodward, 1994). In an aquifer where bioremediation of organic matter is occurring, fermentative microorganisms produce hydrogen as they metabolize the organic matter. Simultaneously, hydrogen is consumed by resperative microorganisms, which use a terminal electronacceptor as an electron sink. The concurrent processes of hydrogen production and hydrogenconsumption produce a steady state concentration of dissolved hydrogen, the magnitude of which depends on the efficiency of the predominant terminal electron acceptor in the process. The more efficient the terminal electron acceptor in promoting hydrogen consumption, the lower the steady state hydrogen concentration. Therefore, hydrogen concentration in a given aquifer (when consid-ered in conjunction with other information such as depletion of terminal electron acceptors and accumulation of reduced products) is a reliable indicator of which TEAP predominates (Lovley, and Goodwin, 1988; Chapelle et al., 1996). Table 1 indicates the relationship between measuredhydrogen concentration and the predominant TEAP in anaerobic conditions (Chapelle et al., 1995). Once a site is characterized in regards to the terminal electron-accepting processes taking place, decisions can be made as to what treatments are appropriate and models can be devised to predict the fate of contaminants at that site (Wiedemeier et al., 1996; Vroblesky and Chapelle,1994).

Use of the Bubble Strip Method for determining gaseous concentrations in well water. The Bubble Strip Method is a chemical testing method developed for the purpose of determining dissolved hydrogen concentrations in well water (Chapelle et al., 1997). The method is based on the principle that gases will undergo a partitioning between a vapor phase and a liquid phase that are in contact with each other. At equilibrium, the partitioning of a gas between a vapor and liquid phase can be quantified by applying Henry's Law. Different gases have characteristic, temperature-dependant Henry's law constants in a given solvent. The stripping procedure involves filling a gas sample bulb with the aqueous solution beinganalyzed and then charging it with 20 mL of air to produce a headspace (Figure 1). The bulb is positioned at a 45° angle to horizontal, and a peristaltic pump upstream of the bulb is used to pump the solution through the bulb such that a stream of water flows through the headspace and produces agitation in the

aqueous phase. During the flowing process, a partitioning of dissolved gases between the two phases occurs, and equilibrium is eventually attained.

Gas chromatographic analysis of the headspace and subsequent application of Henry's Law enables the determination of gaseous concentration in the aqueous solution. In this study, we employ a specially designed system to determine the optimum flow rate at which water should be pumped through the gas sample bulb. We also determine the minimum flow time required for equilibrium to be established in the process of stripping aqueous solutions of each of the three gases being investigated. The effect of varying temperature on time to equilibrium in the stripping of hydrogen is also investigated.

3.0 METHODS AND MATERIALS

Pressure and temperature measurements - A digital manometer (Dwyer Series 475 Mark III) was used to measure differential pressure between the gas sample bulb headspace and the atmosphere during the stripping process. A digital barometer (Fisherbrand) was used to measure barometric pressure. Pressure was not corrected for altitude. Temperature was also displayed on this device.

Instrumentation
Analysis of hydrogen was accomplished using a Trace Analytical RGA3 Reduction Gas Analyzer. Injection volume was 2-mL. The column temperature was set at 100°C and the detector temperature was set at 265°C. The retention time of H2 was 0.50 minutes with a carrier gas (High Purity Nitrogen) flow rate of 22 mL/min.

Methane was analyzed using a Finnigan 9001 Gas Chromatograph equipped with a 1/8-inch, 6-foot, stainless steel, packed-column (Porapak N), and FID detector. Injection volume was 200-µL. The injector temperature was set at 175°C; the column temperature was set at 80°C; and the detector temperature was set at 200°C. The retention time of methane was 0.490 minutes with a carrier gas (High Purity Helium) flow rate of 25 mL/min.

Vinyl chloride was analyzed using a Finnigan 9001 Gas Chromatograph equipped with a 1/8-inch, 6-foot, stainless steel, packed-column (Porapak N), and FID detector. Injection volume was 200 µL. The injector temperature was set at 175°C, the column temperature was set at 120°C, and the detector temperature was set at 200°C. The retention time of vinyl chloride was 2.85 minutes with a carrier gas (High Purity Helium) flow rate of 25 mL/min. For very low concentra-tions of vinyl chloride, an ECD or HECD detector may be preferred over an FID detector.

All injections were made with appropriately sized Pressure-Lok gas tight syringes (Precision Sampling Corporation).

Gas standards - Hydrogen, methane, and vinyl chloride standards were prepared using gases and gaseous mixtures purchased from Scott Specialty Gases.

Preparation of calibration curves

Hydrogen Standard samples were prepared by diluting a hydrogen mixture (100 ppm in nitrogen) into sealed serum bottles (Wheaton) containing nitrogen. Serum bottles were prepared by placing themin a container filled with water purified by reverse osmosis. The bottles were completely filled with water and inverted such that nitrogen gas could be bubbled into them. After the bottles were filledwith nitrogen, they were sealed with a rubber septum (Wheaton, 13 mm x 20 mm Gray butylTeflon-faced, straight Plug Style). The sealed bottles were removed from the water and tear away aluminum seals (Supelco) were applied to secure the septa. Standards were then prepared by withdrawing from the bottles an amount of nitrogen equal to the amount of 100 ppm hydrogen mixture to be added (Table 2). The appropriate amount of 100 ppm hydrogen was then injected and the bottles were allowed to sit for 1 hour before gas chromatographic analysis was attempted. Gas tight syringes were used throughout the procedure.

Methane - Three standards were used to prepare a calibration curve for methane: 100 ppm, 50 ppm, and10 ppm methane in nitrogen. The 100 ppm standard was withdrawn directly from a gas cylinder. The 50 ppm and 10 ppm standards were prepared by diluting 100 ppm methane using the same technique as described above for the hydrogen standards.

Vinyl chloride - Five standards were used to prepare a calibration curve for vinyl chloride: 10 ppm, 100 ppm, 255 ppm, 510 ppm, and 1020 ppm vinyl chloride in nitrogen. The 10 ppm, 100 ppm, and 1020 ppm standards were withdrawn directly from gas cylinders. The 510 ppm and 255 ppm standards were prepared by diluting the 1020 ppm vinyl chloride using the same technique as described above for the hydrogen standards.

Equilibration studies apparatus - A gas sample bulb, 250 mL (Supelco, Inc.), with thermogreen LB-1 cylindrical, half-hole type septa (Supelco, Inc.) was positioned down field of a peristaltic pump (Masterflex, Model 7518-12), such that water could be pumped from a reservoir (Pyrex, 13.45 L), through the gas sample bulb, and back to the reservoir. Another peristaltic pump was used to circulate reservoir headspace through the aqueous solution in order to maintain a constant concentration of gas in the solution during the stripping process. Masterflex tubing (6424-15) was employed in assembling the systemalong with assorted other tubes of various makes and sizes (Figure 2).

Preparing solutions for stripping - The following summarizes the procedures used to prepare solutions for stripping. Separate experiments were done for each of the three gases. To produce a headspace concentration of approximately 10 ppm H2 in the reservoir, reverse osmosis water was pumped into the reservoir until the reservoir had a headspace of 4.5 L. Thesample bulb was also completely filled with water in the process. Hydrogen gas (500 mL, 100 ppmin nitrogen) was injected into the chamber, after which 500 mL of water was released. The headspace was then sparged through the water in the reservoir for 24 hours. Simultaneously, water was circulated through the bulb and reservoir in order to achieve mixing in solution. To produce a reservoir headspace concentration of approximately 100 ppm methane, the reservoir was filled with reverse osmosis water as described above so that there was a 5 L headspace. Methane gas (0.5 mL) was then injected into the chamber headspace, followed by sparging and water circulation for 24 hours. To produce a reservoir headspace of approximately 900 ppm vinyl chloride, the reservoir was filled with reverse osmosis water as described above so that there was

a 5 L headspace. Vinyl chloride gas (4.5 mL) was then injected into the chamber headspace, followed by sparging and water circulation for 24 hours. The headspace concentrations above were significantly lower after equilibrium was established between water and headspace due to the solubility of the gases in water.

Determining time to equilibrium studies were conducted to determine how much stripping time is required for equilibrium to be established between the aqueous and gas phases in the gas sample bulb. The procedure involved subjecting the water from the reservoir to the stripping process over several different time periods (5min., 10 min., 20 min., etc.). For each time period, the gas sample bulb was charged with a new volume of nitrogen gas. On completion of the stripping process over a given time interval, the reservoir and sample bulb headspaces were analyzed by gas chromatography, after which the sample bulb headspace was discharged into the reservoir. Upon injection of a new volume of nitrogen into the gas sample bulb, stripping started over again from time zero. This procedure was repeated until data from all relevant time periods were collected. For use in calculations, differential pressure between the sample bulb headspace and the atmosphere was measured with a digital manometer by way of the septum in the sample bulb during pumping. Additionally, atmospheric pressure (uncorrected for altitude) was recorded during the equilibration studies. Concentrations of the gas in chamber headspace and sample bulb headspace were plotted against flow time. All concentrations were corrected to 1 atm as described in the calculations section. Equilibrium was considered attained when the gaseous concentrations in the chamber and sample bulb headspaces were within 10% of each other.

4.0 CALCULATIONS

Correcting calibration curve data - Areas reported by GC were corrected to standard atmospheric pressure by applying equation1: Area (corrected) = Area (uncorrected) * (29.92 in Hg/Patm)where 29.92 in Hg = standard atmospheric pressure, and Patm = the atmospheric pressure (uncor-rected for altitude). Corrected areas were plotted against concentrations and a line estimation was performed toyield a slope, m, such that gaseous concentration could be calculated directly from peak area (equation 2).[gas] (ppm) = m * area (corrected)where [gas] = the concentration of gas being analyzed, and area (corrected) = the area of thecorresponding peak as reported by GC (corrected to standard atmospheric pressure).

Correcting sample bulb headspace concentrations - When the stripping process was complete, the headspace concentrations were calculated using equation 2, and a correction was made for differential pressure in the sample bulb headspace using equation 3:[gas]headspace(ppm) = [gas] (ppm) * (1 bar + P (bar)/1 bar) where P is the difference in pressure between the headspace in the sample bulb and the ambient atmosphere during the pumping process. Standard atmospheric pressure is approximated as 1 bar.

Concentration of dissolved gas in solution - Concentration of dissolved gas in solution is calculated using equations 4 and 5 (Maron andPrutton, 1965):

X(gas)solution(mol H2/mol H2O) = [gas]headspace(ppm)x10-6atm*[K'(atm-1)] and
[gas]solution (mol/L) = X(gas)solution(mol H2/mol H2O) * 55.345 mol H2O/L(1)(2)(3)(5)(4)

Where X(gas) solution is the mole fraction of dissolved gas in solution (mole H2O » mol gas);[gas]headspace is the concentration of gas in the headspace in ppm as determined by GC analysis and corrected to 1 atmosphere; K' is the Henry's law constant, which differs for different gases and is dependant on temperature; [gas] solution is the concentration of dissolved gas in the aqueous solutionin moles/liter. Henry's constants are recorded for methane at 250C (Manahan, 1994), for hydrogen at various temperatures (Maron and Prutton, 1965), and for vinyl chloride (Lide, 1996).

Sample calculation - For a corrected headspace concentration of 0.50 ppm hydrogen at 40C:X(gas)solution(mol H2/mol H2O) = 0.50 x 10-6atm * 1.72 x 10-5atm-1= 8.6 x 10-12mol H2/mol H2Oand[gas]solution(mol/L) =8.6 x 10-12mol H2/mol H2O * 55.345 mol H2O/L= 4.8 x 10-10mol H2/L H2O = 0.48 nMA hydrogen concentration of 0.50 ppm in the sample bulb headspace translates into a concentrationof 0.48 nM in solution.

5.0 RESULTS

Experiments were done to determine how much stripping time was necessary in order for equilibrium to be achieved between the aqueous and gas phases in the gas sample bulb. Flow rates of 300 mL/minute and 400 mL/minute were employed at room temperature for aqueous solutions of hydrogen (figures 3,4), vinyl chloride (figures 5,6), and methane (figures 7,8). For hydrogen solutions, experiments were also done at 40C (figures 9,10). The results show that at room temperature, equilibrium is achieved within 20 minutes when hydrogen is stripped from water at a rate of 400 mL/minute. This is evidenced by the hydrogen concentration in the sample bulb headspace reaching 90% of that in the reservoir headspace. Concentration of hydrogen in solution (as calculated using reservoir headspace data) was approximately 3 nM. At the same flow rate and temperature, equilibrium is achieved within ten minutes for methane and within five minutes for vinyl chloride, with solution concentrations of 240 nM and 10mM, respectively. At 40C, equilibrium is attained within 30 minutes for an aqueous solution of hydrogen.The slower flow rate of 300 mL/minute resulted in longer times to equilibrium. Flow times of less than 300 mL/minute did not produce the agitation necessary for equilibrium to be attained in a reasonable amount of time. Flow rates of greater than 400 mL/minute resulted in the rapid loss of sample bulb headspace as a result of a large pressure differential which existed between the bulb headspace and the reservoir headspace during pumping.

6.0 CONCLUSION

It is demonstrated in this paper that the Bubble Strip Method is a viable technique for the collection of hydrogen, vinyl chloride and methane from aqueous solution. We show that after the stripping process is complete, nanomolar concentrations of these gases in aqueous solution can be determined by gas chromatographic analysis of sample bulb headspace, followed by application of Henry's law.

Stripping time required for equilibrium to be attained between solution and headspace was determined to be on the order of 20 minutes for aqueous solutions of hydrogen at room temperature. A longer time to equilibrium (30 minutes) was required at 40C.

Aqueous solutions of vinyl chloride and methane reached equilibrium with a headspace after five and ten minutes ofstripping, respectively, at room temperature.

The optimum flow rate for the stripping process was determined to be 400 mL/min. Lower flow rates resulted in longer times to equilibrium, while higher flow rates resulted in significant loss of bubble volume with time due to a large differential pressure between the sample bulb headspace and the reservoir headspace.

Based on this study, the Bubble Strip Method is potentially useful at field sites for quickly obtaining reliable data regarding the concentrations of various gases in well water.

7.0 ACKNOWLEDGMENTS

The authors would like to acknowledge funding support from the Air Force Center for Environmental Excellence at Brooks AFB, Texas (IAG-RW57938459). Although this research was conducted at an U.S. EPA facility, the report has not been subjected to U.S. EPA review. It does not necessarily reflect the views of the agancy and an official endorsement should not be inferred.

8.0 REFERENCES

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9.0 TABLES

Table 1. Hydrogen concentration versus predominant TEAP in anaerobic conditions.
Table 2. Preparation of hydrogen standards.

10.0 FIGURES

Figure 1. A solution-filled gas sample bulb is charged with 20 mL of headspace in preparation forstripping.
Figure 2. Equilibration studies are performed by stripping a solution whose concentration is kept constant.
Figure 3. A comparison of reservoir and sample bulb headspace concentrations of hydrogen as a function of stripping time.
Figure 4. A comparison of reservoir and sample bulb headspace concentrations of hydrogen as a function of stripping time.
Figure 5. A comparison of reservoir and sample bulb headspace concentrations of vinyl chloride as a function of stripping time.
Figure 6. A comparison of reservoir and sample bulb headspace concentrations of vinyl chloride as a function of stripping time.
Figure 7. A comparison of reservoir and sample bulb headspace concentrations of methane as a function of stripping time.
Figure 8. A comparison of reservoir and sample bulb headspace concentrations of methane as afunction of stripping time.
Figure 9. A comparison of reservoir and sample bulb headspace concentrations of hydrogen as afunction of stripping time.

Figure 10. A comparison of reservoir and sample bulb headspace concentrations of hydrogen as afunction of stripping time.

NOTE: Tables and Figures (not included in this document) are available as appendices to this SOP.

STANDARD OPERATING PROCEDURES

No. I-DissolvedOxygen-RC

Dissolved Oxygen
Membrane Electrode Method
(EPA 360.1 and SM 4500-O-G)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Dissolved Oxygen by Membrane Electrode Method

1.0 Scope and Application

1.1 This SOP is based on EPA Method 360.1, and Standard Method 4500-O-G for the determination of dissolved oxygen using the memebrane electrode or probe method.

1.2 The probe method for dissolved oxygen is recommended for those samples containing materials which interfere with the modified Winkler procedure such as sulfite, thiosulfate, polythionate, mercaptans, free chlorine or hypochlorite, organic substances readily hydrolyzed in alkaline solutions, free iodine, intense color or turbidity and biological flocs.

1.3 The probe method is recommended as a substitute for the modified Winkler procedure in monitoring of streams, lakes, outfalls, etc., where it is desired to obtain a continuous record of the dissolved oxygen content of the water under observation.

1.4 The probe method may be used as a substitute for the modified Winkler procedure in BOD determinations where it is desired to perform nondestructive DO measurements of a sample.

1.5 The probe method may be used under any circumstances as a substitute for the modified Winkler procedure provided that the probe itself is standardized against the Winkler method on samples free of interfering materials.

1.6 The electronic readout meter for the output from dissolved oxygen probes is normally calibrated in convenient scale (0 to 10, 0 to 15, 0 to 20 mg/L for example) with a sensitivity of approximately 0.05 mg/L.

2.0 Summary of Method

2.1 The most common instrumental probes for determination of dissolved oxygen in water are dependent upon electrochemical reaction. Under steady-state conditions, the current or potential can be correlated with DO concentrations. Interfacial dynamics at the probe-sample interface are a factor in probe response and a significant degree of interfacial turbulence is necessary. For precision performance, turbulence should be constant.

3.0 Interferences

3.1 Dissolved organic materials are not known to interfere in the output from dissolved oxygen probes.

3.2 Dissolved oxygen probes are temperature sensitive, and temperature compensation is normally provided by the manufacturer. Membrane probes have a temperature coefficient of 4 to 6 % / ^{o}C dependent upon the membrane employed.

3.3 Dissolved inorganic salts are a factor in the performance of dissolved oxygen probe.

3.3.1 Probes with membranes respond to partial pressure of oxygen, which in turn is a function of dissolved inorganic salts. Conversion factors for seawater and

brackish waters may be calculated from dissolved oxygen saturation versus salinity data.

3.3.2 Conversion factors for specific organic salts may be developed experimentally. Broad variations in the kinds and concentrations of salts in samples can make the use a membrane probe difficult.

3.4 Reactive compounds can interfere with the output or the performance of dissolved oxygen probes.

3.4.1 Reactive gases, which pass through the membrane probes, may interfere. For example, chlorine will depolarize the cathode and cause a high probe-output. Long-term exposures to chlorine will coat the anode with the chloride of the anode metal and eventually desensitize the probe. Alkaline samples in which free chlorine does not exist will not interfere.

3.4.2 Hydrogen sulfide will interfere with membrane probes if the applied potential is greater than the half-wave potential of the sulfide ion. If the applied potential is less than the half-wave potential, an interfering reaction will not occur, but coating of the anode with the sulfide of the anode metal can take place.

4.0 Apparatus

4.1 No specific probe or accessory is especially recommended as superior. However, probes which have been evaluated or are in use and found to be reliable are the Weston & Stack DO Analyzer Model 30, the Yellow Springs Instrument (YSI) Model 54, and the Beckman Field lab Oxygen Analyzer.

5.0 Sample Collection, Preservation, and Handling

5.1 Oblong 4-Oz HDPE bottles (Level 6 precleaned) with foamed polyethylene lined polypropylene screw cap closures may be used. Provide one bottle for each sample to be collected. Preservatives are not used.

5.2 This test is normally performed in the field to characterize groundwater, surface water or waste water. Collect samples with minimal headspace to reduce changes to the dissolved oxygen concentration.

5.3 Samples should be analyzed upon receipt in the laboratory.

6.0 Health, Safety, and Waste Disposal

6.1 Samples may contain high level of organic and inorganic contaminants. Gloves and safety glasses should be worn to minimize contact of the sample with skin and eyes.

6.2 Toxicity of the samples is not dependent on the oxygen content. Dispose of unused samples in accordance with AML waste disposal policies and procedures.

7.0 Calibration

Several dissolved oxygen meters are used in the laboratory. Follow manufacturer instructions for instrument calibration. Generally, calibrate membrane elcetrodes by reading against air or a water sample saturated with air.

8.0 Procedure

8.1 Follow manufacturer instructions for sample measurements. Take care in changing membrane to avoid contamination of the sensing element and also trapping of minute air bubbles under the membrane, which can lead to lower response and high residual current.

8.2 Provide sufficient sample flow across membrane surface to overcome erratic response. Stirring is recommended during calibration and sample analysis.

9.0 Calculations

9.1 Use procedures recommended by the manufacturer. An accuracy of ±0.1-mg/L of DO with a precision of ±0.05-mg/L is possible with the memebrane electrode method.

9.2 Report results as dissolved oxygen in mg/L at 25°C.

10.0References

10.1Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Dissolved Oxygen, Method 360.1 (Membrane Electrode), Issued 1971.

10.2Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Oxygen, Method 4500-O and 4500-O G, Membrane Electrode Method.

10.3AML Standard Operating Procedures, Q-Water-RC.

10.4AML Standard Operating Procedures, Q-WasteMgmt-RD.

STANDARD OPERATING PROCEDURES

No. I-Hardness-RC

Hardness and Calcium Hardness (SM 2340B)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Total Hardness (SM 2340B)

1.0 Scope and Application

1.1 This standard operating procedure (SOP) is based on Standard Method (SM) 2340B, a computational procedure for the determination of total hardness and/or calcium hardness in mg/L of calcium carbonate.

1.2 This SOP is suitable for all concentration ranges of calcium and magnesium that are determinable by ICP (EPA 200.7 or SW846 6010B). This SOP is applicable to drinking, surface, saline, and ground water.

2.0 Summary of Method

2.1 Calcium and magnesium in the water sample are determined in mg/L using ICP-based determinative procedures such as EPA 200.7 or SW846 6010B.

2.2 The total hardness in mg/l of $CaCO_3$ is calculated from the Ca and Mg results.

3.0 Interferences

3.1 For specific interferences to the calcium and magnesium, see determinative ICP-AES procedures for their measurement.

4.0 Sample Handling and Preservation

4.1 See SOPs based on EPA 200.7 and/or SW-846 6010B.

5.0 Calculations

5.1 Calculate total hardness in mg/l $CaCO_3$ from independently determined amounts (mg/L) of calcium and magnesium:

Total Hardness (mg $CaCO_3$/L) = (2.497 * Amt. Of Ca) + (4.118 * Amt. of Mg)

5.2 Calculate calcium hardness in mg/l $CaCO_3$ from independently determined amount (mg/L) of calcium:

Ca Hardness (mg $CaCO_3$/L) = (2.497 * Amt. Of Ca)

6.0 References

6.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Hardness.
6.2 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Acidity, Method 2340B.
6.3 AML Standard Operating Procedures, Q-Water-RC.
6.4 AML Standard Operating Procedures, Q-WasteMgmt-RD.
6.5 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
6.6 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-Nitrate-Nitrite-353-354-RC

Nitrate and Nitrite by Manual Cadmium Reduction and Spectrophotometry (EPA 353.3, 354.1, SM 4500-NO_2^-, and SM 4500-NO_3^-)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

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Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Nitrate and Nitrite by Mnaual Cadmium Reduction and Spectrophotometry (EPA 353.3, 354.1, SM 4500-NO_2^-, and SM 4500-NO_3^-)

1.0 Scope and Application

1.1 This SOP is based on EPA Method 353.3, 354.1, and Standard Method 4500-NO_2^--A abd -B, 4500-NO_3^--A and -E for the determination of nitrite and nitrate after cadmium reduction by spectrophotometry.
1.2 This method is applicable to the determination of nitrite singly, or nitrite and nitrate combined in drinking, surface and saline waters, domestic and industrial wastes.
1.3 The applicable range of this method is 0.01 to 1.0 mg/L nitrate-nitrite nitrogen. The range may be extended with sample dilution.

2.0 Summary of Method

2.1 A filtered sample is passed through a column containing granulated copper-cadmium to reduce nitrate to nitrite. The nitrite (that originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye which is measured spectrophotometrically.
2.2 Separate, rather than combined nitrate-nitrite, values are readily obtained by carrying out the procedure first with, and then without, the Cu-Cd reduction step.

3.0 Sample Handling and Preservation

3.1 Analysis should be made as soon as possible. If analysis can be made within 24 hours, the sample should be preserved by refrigeration at 4 ^oC. When samples must be stored for more than 24 hours, they should be preserved with sulfuric acid (2 mL H_2SO_4 per Liter) and refrigeration.

Caution: Samples for reduction column must not be preserved with mercuric chloride.

4.0 Interferences

4.1 Build up of suspended matter in the reduction column will restrict sample flow. Since nitrate-nitrogen is found in a soluble state, the sample may be pre-filtered through a glass fiber filter or a 0.45 u membrane filter. Highly turbid samples may be pretreated with zinc sulfate before filtration to remove the bulk of particulate matter present in the sample
4.2 Low results might be obtained for samples that contain high concentrations of iron, copper or other metals. EDTA is added to the samples to eliminate this interfere.
4.3 Samples that contain large concentrations of oil and grease will coat the surface of the cadmium. This interference is eliminated by pre-extracting the sample with an organic solvent.

4.4 This procedure determines both nitrate and nitrite. If only nitrate is desired, a separate determination must be mode for nitrite and subsequent corrections made. The nitrite may be determined by the procedure below without the reduction step.

5.0 Apparatus

5.1 Reduction column: The column in Figure I was constructed from a 100 mL pipet by removing the top portion. This column may also be constructed from two pieces of tubing joined end to end. A 10 mm length of 3 cm I.D. tubing is joined to a 25 cm length of 3.5 mm I.D. tubing.

5.2 Spectrophotometer for use at 540 nm, providing a light path of 1 cm of longer.

6.0 Reagents

6.1 Granulated cadmium: 40-60 mesh (MCB Reagents).

6.2 Copper-Cadmium: The cadmium granules (new or used) are cleaned with dilute HCL and copperized with 2% solution of copper sulfate in the following manner:

6.2.1 Wash the cadmium with dilute HCL (6.10) and rinse with distilled water. The color of the cadmium should be silver.

6.2.2 Swirl 25 g cadmium in 100 mL portions of a 2% solution of copper sulfate (6.11) for 5 minutes or until blue color partially fades, decant and repeat with fresh copper sulfate until a brown colloidal precipitate forms.

6.2.3 Wash the copper-cadmium with distilled water (at least 10 times) to remove all the precipitated copper. The color of the cadmium so treated should be black.

6.3 Preparation of reaction column: Insert a glass wool plug into the bottom of the reduction column and fill with distilled water. Add sufficient copper-cadmium granules to produce a column 18.5 cm in length. Maintain a level of distilled water above the copper-cadmium granules to eliminate entrapment of air. Wash the column with 200 mL of dilute ammonium chloride solution (6.5). The column is then activated by passing through the column 100 mL of a solution composed of 25 mL of a 1.0 mg/L NO_3-N standard and 75 mL of ammonium chloride-EDTA solution (6.4). Use a flow rate between 7 and 10 mL per munite.

6.4 Ammonium chloride - EDTA solution: Dissolve 13 g ammonium chloride and 1.7 g disodium ethylenediamine tetracetate in 900 mL of distilled water. Adjust the pH to 8.5 with conc. Ammonium hydroxide (6.9) and dilute to 1 Liter.

6.5 Dilute ammonium chloride-EDTA solution: Dilute 300 mL of ammonium chloride-EDTA solution (6.4) to 500 mL with distilled water.

6.6 Color reagent: Dissolve 10 g sulfanilamide and 1 g N(1-naphthyl)-ethylene-diamine dihydrochloride in a mixture of 100 mL conc. Phosphoric acid and 800 mL of distilled water and dilute to 1 Liter with distilled water.

6.7 Zinc sulfate solution: Dissolve 100 g $ZnSO_4 \bullet 7H_2O$ in distilled water and dilute to 1 Liter.

6.8 Sodium Hydroxide Solution, 6N: Dissolve 240g NaOH in 500 mL distilled water, cool and dilute to 1 Liter.

6.9 Ammonium Hydroxide, conc.

6.10 Dilute hydrochloric acid, 6N: Dilute 50 mL of conc. HCl to 100 mL with distilled water.

6.11 Copper Sulfate Solution, 2%: Dissolve 20 g of $CuSO_4 \bullet 5H_2O$ in 500 mL of distilled water and dilute to 1 Liter.

6.12 Stock Nitrate Solution: Dissolve 7.218 g KNO_3 in distilled water and dilute to 1000 mL. Preserve with 2 mL of chloroform p[er liter. This solution is stable for at least 6 months. 1.0 mL = 1.00 mg NO_3-N.

6.13 Standard Nitrate Solution (LCS): Dilute 10.0 mL of nitrate stock solution (6.12) to 1000 mL with distilled water. 1.0 mL = 0.01 mg NO_3-N. (1000 mg/L). Prepare LCS for a final concentration of 0.3 mg/L by adding 0.03ml of standard to 100 mL.

6.14 Stock Nitrite Solution: Dissolve 6.072 g KNO_2 in 500 mL of distilled water and dilute to 1000 mL. Preserve with 2 mL of chloroform and keep under refrigeration. Stable for approximately 3 months. 1.0 ml = 1.00 mg NO_2-N.

6.15 Standard nitrite solution (LCS): Dilute 10.0 mL of nitrite stock solution (6.14) to 1000 mL with distilled water. 1.0 mL = 0.01 mg NO_2-N. (1000 mg/L). Prepare LCS for a final concentration of 0.3 mg/L by adding 0.03ml of standard to 100 mL.

6.16 Purchase a prepared, certified 1000 mg/L standard Nitrate solution.

6.17 Purchase a prepared, certified 1000 mg/L standard Nitrite soltuion.

6.18 Using standard nitrate solution (6.16) prepare the following standards in 100 mL volumetric flasks:

Concentration mg NO_3-N / L	mL of Standard Solution / 100 mL
0.00	0.00
0.10	0.01
0.20	0.02
0.50	0.05
1.00	0.10

7.0 Procedure

7.1 Turbidity removal: One of the following methods may be used to remove suspended matter.

7.1.1 Filter sample through a glass fiber filter or a 0.45 *u* membrane filter.

7.1.2 Add 1 mL zinc sulfate solution (6.7) to 100 mL of sample and mix thoroughly. Add 0.4-0.5 mL sodium hydroxide solution (6.8) to obtain a pH of 10.5 as determined with a pH meter. Let the treated sample stand a few minutes to allow the heavy flocculent precipitate to settle. Clarify by filtering through a glass fiber filter of a 0.45 *u* membrane filter.

7.2 Oil and grease removal: Adjust the pH of 100 mL of filtered sample to 2 by addition of conc. HCl. Extract the oil and grease from the aqueous solution with two 25 mL portions of a non-polar solvent (Hexane).

7.3 If the pH of the sample is below 5 or above 9, adjust to between 5 and 9 with either conc. HCl or conc. HN_4OH. This is done to insure a sample pH of 8.5 after step 7.4.

7.4 To 25.0 mL of sample or an aliquot diluted to 25.0 mL, add 75 mL of ammonium chloride-EDTA solution (6.4) and mix.

7.5 Pour sample in to column and collect sample at a rate of 7-10 mL per minute/

7.6 Discard the first 25 mL, collect the rest of the sample (approximately 70 mL) in the original sample flask. Reduced samples should not be allowed to stand longer than 15 minutes before addition of color reagent, step 7.7.

7.7 Add 2.0 mL of color reagent (6.6) to 50 mL of sample. Allow 10 minutes for color development. Within 2 hours measure the absorbance at 540 nm against a reagent blank. NOTE: If the concentration of sample exceeds 1.0 mg NO3-N / L, the remainder of the reduced sample may be used to make an appropriate dilution before proceeding with step 7.7.

7.8 Standards: Carry out the reduction of standards exactly as described for the samples. At least one nitrite standard should be compared to a reduced nitrate standard at the same concentration to verify the efficiency of the reduction column.

8.0 Calculation

8.1 Obtain a standard curve by plotting the absorbance of standards run by the above procedure against NO_3-N mg/L. Compute concentration of samples by comparing sample absorbance with standard curve.

9.0 Quality Control

9.1 A Method Blank should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

9.2 A Second Source Standard (LCS) should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

9.3 A Sample Duplicate should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

9.4 The correlation coefficient of the standard curve must be greater than or equal to 0.995.

10.0References

10.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Nitrogen, Nitrate-Nitrite, Method 353.3, Spectrophotometric, Cadmium Reduction, Issued, 1974.

10.2 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Nitrogen, Nitrite, Method 354.1, Spectrophotometric, Issued, 1971.

10.3 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Method 4500-NO_2^--A and 4500-NO_2^--B.

10.4 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Method 4500-NO_3^--A and 4500-NO_3^--E.

10.5 AML Standard Operating Procedures, Q-Water-RC.

10.6 AML Standard Operating Procedures, Q-WasteMgmt-RD.

10.7 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

10.8 AML Standard Operating Procedures, Q-ControlCharts-RC.

10.9

STANDARD OPERATING PROCEDURES

No. I-Nitrocellulose-Soil-RC

NITROCELLULOSE IN SOIL

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) is based on the United States Army Engineer Research and Development (USA-CRREL) procedure, which is based on an earlier USATHAMA Method for the determination of trace levels of nitrocellulose (NC) in water by ion chromatography. Samples need ultra-filtration, digestion, extraction, and cleanup procedures prior to instrumental analysis.

1.2 All analysts performing this method should have 6-months prior experience in performing wet chemistry analysis including the analysis of nitrate and nitrite by ion chromatography and/or spectrophotometry.

1.3 The sample extraction and cleanup procedures described in this SOP should be followed strictly as written. Any modifications to this SOP should be reviewed and approved by the supervisor and QA Director prior to implementation and incorporation into the SOP.

1.4 The estimated reporting limit of the CCREL method is 73-µg/L. The linear concentration range was calculated as 4 to 830-µg/L NC. The range may be extended with appropriate dilution of the aqueous sample digestates.

2.0 SUMMARY OF METHOD

2.1 The nitrocellulose (NC), normally present in water in particulate form is isolated from water samples by vacuum filtration through a 0.02-µm Anapore inorganic membrane. The membrane is washed with methanol to remove other interfering esters such as PETN.

2.2 The filter membrane is then soaked in acetone to extract NC. The acetone extract is evaporated to dryness and the residue containing nitrocellulose is hydrolyzed with 1N NaOH at 100°C for thirty minutes to liberate nitrate and nitrite.

2.3 The caustic soda is neutralized by passage through a cation exchange sample preparation cartridge in the hydrogen form.

2.4 The liberated nitrates and nitrites are determined by ion chromatography.

2.5 The NC concentration is calculated from the mass of nitrogen found as nitrate and nitrite.

3.0 INTERFERENCES

3.1 Soil and sediment samples may contain high levels of inorganic nitrate and nitrite, which are removed by aqueous extraction procedures described in this SOP. Since nitrocellulose is determined from the nitrate/nitrite content in organic solvent extracts, incomplete removal of inorganic nitrate prior to solvent extraction may result in positive bias for nitrocellulose.

3.2 Interferences may also be caused by other nitrate/nitrite containing organic compounds (e.g. explosives) that are insoluble in water and extractable by acetone. A methanol wash of the sample may be used to either reduce or to remove some of these organic interferences. However, methanol was procedure has not been evaluated for routine application.

3.3 Solvents, reagents, glassware can produce laboratory-generated artifacts, and background interferences, which can produce false positive results. A method blank

(MB) analysis is used to verify the absence of these contaminants. If the MB contains the target analyte at or above one half of the practical or method quantitation limit (PQL or MQL), appropriate corrective action may be necessary.

3.4 Build up of suspended matter in the cadmium reduction column will restrict sample flow. Since nitrate-nitrogen is found in a soluble state, the sample may be pre-filtered through a glass fiber filter or a 0.45-μ membrane filter. Highly turbid samples may be pretreated with zinc sulfate before filtration to remove the bulk of particulate matter present in the sample.

3.5 Sample results exceeding site specific action level should be carefully evaluated to determine if potential interferences exist. Recommendations should be made to the Client Project Manager to perform additional analyses such as the analysis for nitroaromatic explosives (SW-846 Method 8330 and 8332) and inorganic nitrate and nitrite (EPA 300.0, and/or 353.3). The laboratory should not perform such additional analyses without prior approval by the Client Project Manager.

3.6 All glassware used during the extraction procedures is washed using the following procedure:

3.6.1 Soak glassware in hot soap and water
3.6.2 Scrub using a laboratory brush
3.6.3 Rinse with tap water
3.6.4 Rinse with deionized water 3 times
3.6.5 Rinse with acetone and dry
3.6.6 Store glassware in a clean area.

4.0 APPARATUS AND MATERIALS

4.1 Centrifuge, Fisher Scientific Model Marathon 3200. See instrument manual for instrument operating conditions.

4.2 Ultrasonic Bath, Branson Model 8210. Maintain at room temperature or less by cycling tap water through the bath during sample extraction. Consult with analysts experienced in explosive analysis in soil by SW-846 Method 8330 for detailed operation procedures. See instrument manual for instrument operating conditions.

4.3 Equipment (home-built) for air-drying soil samples for NC and explosives analysis.

4.4 VOA vials (40 ml) with Teflon lined caps.

4.5 Vortex Genie

4.6 Water Bath

4.7 Wrist Action Shaker

4.8 Cadmium Reduction Columns

4.9 Hamilton syringes- various sizes

4.10 Vacuum Desiccator

4.11 Volumetric flasks-Class A- various size

4.12 Air drying oven

4.13 Spectrophotometer, Milton Roy, Model Spectronic 20. See instrument manual for instrument operating conditions. Use at 540-nm, providing a light path of 1-cm or longer.

4.14 Cadmium reduction column: The column (see Appendix 1 for details) was constructed from a 100-ml pipette by removing the top portion. This column may also be

constructed from two pieces of tubing joined end to end. A 10-mm length of 3-cm I.D. tubing is joined to a 25-cm length of 3.5-mm I.D. tubing.

5.0 REAGENTS

All chemicals used are ACS Reagent grade or better. If contamination is found at or above method detection limit, all suspected chemicals are screened individually and replaced.

5.1 The following reagents are used with the NC determination part of the method:

- 5.1.1 Reagent water: Filtered (0.45 μ), Type I water from USFilter water system.
- 5.1.2 Acetone - Pesticide or HPLC grade (EM Science or equivalent). Acetone is extremely flammable. All procedures involving transfer of acetone solutions should be performed in a hood.
- 5.1.3 Methanol – Pesticide or HPLC grade (EM Science or equivalent). Methanol is extremely flammable. All procedures involving transfer of acetone solutions should be performed in a hood.
- 5.1.4 Sodium Hydroxide, Fisher Scientific, ACS Reagent grade. Sodium hydroxide is corrosive.
- 5.1.5 Sulfuric Acid, concentrated, Fisher Scientific, ACS Reagent grade. Acids are corrosive.
- 5.1.6 Nitrocellulose, Aldrich cat # 43503-1 containing 12% nitrogen. Nitrocellulose (NC) is shipped with isopropanol to minimize the chance of combustion. Prior to preparing stock solutions, prepare this reagent by spreading approximately 1-g evenly over the bottom of a weighing boat. Place the weighing boat in a desiccator for several days to eliminate the isopropanol and to dry the nitrocellulose. Use the dried nitrocellulose to prepare stock standards.
- 5.1.7 Laboratory sand. Pre-wash with Type II water, followed by Type I water, dry in oven and store in an appropriately labeled, clean, glass container.
- 5.1.8 Sodium hydroxide (1N) solution - Dissolve 40-g of NaOH in 1000-ml of Type I or II water, mix well, and transfer to a properly labeled plastic container.
- 5.1.9 Sulfuric acid (1N) solution - Add 28-ml of concentrated H_2SO_4 to 500-ml of Type I or II reagent water in a 1000-ml volumetric. Dilute to volume with reagent water, mix well, and transfer to a properly labeled plastic container.

5.2 The following reagents are used with the cadmium reduction and nitrite determination part of the method:

- 5.2.1 Granular cadmium, 40-60 mesh, Fisher Scientific, ACS reagent grade.
- 5.2.2 Copper sulfate ($CuSO_4.5H_2O$), Fisher Scientific, ACS reagent grade.
- 5.2.3 Potassium nitrate, Fisher Scientific, ACS reagent grade.
- 5.2.4 Hydrochloric acid, concentrated, Fisher Scientific, ACS reagent grade.
- 5.2.5 Phosphoric acid, concentrated, Fisher Scientific, ACS reagent grade.
- 5.2.6 Sulfanilamide, Fisher Scientific, ACS reagent grade.
- 5.2.7 N(1-napthyl)-ethylenediamine dihydrochloride, Fisher Scientific, ACS reagent grade.
- 5.2.8 Ammonium chloride, Fisher Scientific, ACS reagent grade.
- 5.2.9 Disodium ethylenediamine tetracetate (EDTA), Fisher Scientific, ACS reagent grade.

5.2.10 Zinc Sulfate ($ZnSO_4.7H_2O$), $CuSO_4.5H_2O$, Fisher Scientific, ACS reagent grade.

5.2.11 Ammonium Hydroxide, concentrated, Fisher Scientific, ACS reagent grade.

5.2.12 Dilute hydrochloric acid, 6N: Dilute 50-ml of conc. HCl to 100-ml with Type I or II reagent water.

5.2.13 Copper Sulfate Solution, 2% - Dissolve 20-g of $CuSO_4 \bullet 5H_2O$ in 500-ml of reagent water and dilute to 1-liter.

5.2.14 Ammonium chloride - EDTA solution: Dissolve 13-g ammonium chloride and 1.7-g of EDTA in 900-ml of reagent water. Adjust the pH to 8.5 with conc. ammonium hydroxide and dilute to 1-liter with reagent water.

5.2.15 Dilute ammonium chloride-EDTA solution: Dilute 300-ml of ammonium chloride-EDTA solution from the previous step to 500- ml with distilled water.

5.2.16 Color reagent: Dissolve 10-g sulfanilamide and 1-g N(1-naphthyl)-ethylene-diamine dihydrochloride in a mixture of 100-mL conc. phosphoric acid and 800-ml reagent water. Dilute to 1-liter with reagent water.

5.2.17 Zinc sulfate solution: Dissolve 100 g $ZnSO_4 \bullet 7H_2O$ in distilled water and dilute to 1 Liter.

5.2.18 Sodium Hydroxide Solution, 6N: Dissolve 240-g NaOH in 500- ml reagent water, cool and dilute to 1-liter.

5.2.19 Copper-Cadmium Reagent: The cadmium granules (new or used) are cleaned with dilute HCL and copperized with 2% solution of copper sulfate in the following manner: Wash the cadmium with dilute HCL (Section 5.2.12) and rinse with reagent water. The color of the cadmium should be silver. Swirl 25-g cadmium (Section 5.2.1) in 100-ml portions of a 2% solution of copper sulfate (Section 5.2.13) for 5-minutes or until blue color partially fades, decant and repeat with fresh copper sulfate until a brown colloidal precipitate forms. Wash the copper-cadmium with reagent at least 10-times to remove all the precipitated copper. The color of the cadmium so treated should be black.

5.2.20 Copper-Cadmium Reduction Column: Insert a glass wool plug into the bottom of the reduction column and fill with reagent water. Add sufficient copper-cadmium granules to produce a column 18.5-cm in length. Maintain a level of distilled water above the copper-cadmium granules to eliminate entrapment of air. Wash the column with 200-ml of dilute ammonium chloride-EDTA solution (Section 5.2.15). The column is then activated by passing through the column 100-ml of a solution composed of 25-ml of a 1.0-mg/L NO_3-N standard and 75-ml of ammonium chloride-EDTA solution (Section 5.2.14). Use a flow rate between 7- and 10-ml per minute.

5.2.21 Hexane - ACS Reagent grade (EM Science or equivalent). Hexane is extremely flammable. All procedures involving transfer of hexane solutions should be performed in a hood.

5.3 Stock Standard Solutions. Prepare stock solutions of nitrocellulose, inorganic nitrate and inorganic nitrite to be used in various steps.

5.3.1 Nitrocellulose (NC) Stock Standard Solution (1000-ug/ml in acetone). Weigh 0.100-g of the dried nitrocellulose (See 5.1.4) into a 100-ml volumetric. Add approximately 50-ml of acetone to dissolve the NC. Dilute to volume with acetone and mix well. Transfer to a properly labeled 125-ml glass bottle and store at 4°C . The stock

standard solution expires in 1-year from the date of preparation. Replace sooner if signs of degradation are observed.

5.3.2 Stock Nitrate Solution: Dissolve 7.218-g KNO_3 (Section 5.2.3) in reagent water and dilute to 1000-mL. Preserve with 2-ml of chloroform per liter. This solution (1000-mg/L NO_3-N) is stable for at least 6-months. 1.0-mL = 1.00-mg NO_3-N.

5.3.3 Standard Nitrate Solution: Dilute 10.0-ml of nitrate stock solution (Section 5.3.2) to 1000-ml with reagent water. 1.0-ml = 0.01-mg NO_3-N. Prepare this solution (10-mg/L NO_3-N) fresh daily.

5.3.4 Purchase a prepared, certified 1000-mg/L standard Nitrate solution from suitable vendors (e.g. Ultra Scientific)

5.3.5 Purchase a prepared, certified 1000-mg/L standard Nitrite solution (e.g. Ultra Scientific).

5.4 Nitrate Calibration Standards - These are prepared fresh daily from the 10-mg/L NO_3-N standard nitrate solution (Section 5.3.3). Six solutions consisting of one calibration blank and five nitrate concentrations are prepared by diluting the standard nitrate solution with reagent water (Section 5.1.1).

5.4.1 Concentration (mg/L) of NO_3-N = 0.00: Dilute 0.00-ml of the standard nitrate solution to 100-ml with reagent water.

5.4.2 Concentration (mg/L) of NO_3-N = 0.05: Dilute 0.50-ml of the standard nitrate solution to 100-ml with reagent water.

5.4.3 Concentration (mg/L) of NO_3-N = 0.10: Dilute 1.00-ml of the standard nitrate solution to 100-ml with reagent water.

5.4.4 Concentration (mg/L) of NO_3-N = 0.20: Dilute 2.00-ml of the standard nitrate solution to 100-ml with reagent water.

5.4.5 Concentration (mg/L) of NO_3-N = 0.50: Dilute 5.00-ml of the standard nitrate solution to 100-ml with reagent water.

5.4.6 Concentration (mg/L) of NO_3-N = 1.00: Dilute 10.0-ml of the standard nitrate solution to 100-ml with reagent water.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 One 4-Oz or 8-Oz wide mouthed sample jar with Teflon lined screw caps may be used for collecting soil, sediment and waste samples. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Quality control samples such as MS and MSD should be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. These samples may be shared with samples collected for other analyses such as explosives (8330), semivolatiles (8270C), PCBs (8082), Diesel (DRO, 8015-mod.) and pesticides (8081, 8140, etc.).

6.2 The soil samples must be air-dried, ground, and sieved (30-mesh) if necessary prior to analysis. Air-dried soil samples cannot be used for other tests other than explosives and nitroglycerin analysis by SW-846 Method 8330. Samples must be air-dried and extracted within 14 days of sample collection dates.

6.3 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Holding times may not have been established for this unusual parameter. Due to the high stability of the nitrocellulose, the holding time concept may not even be applicable. Analytical data

generated outside of the specified holding times, if any should be considered to be minimum values only. The laboratory holding time for the extraction of soil, sediment and waste samples, which includes TCLP leachate preparation is 14 days from the date of sample collection or receipt. Samples extracts are stored in amber bottles and/or vials at 4°C just prior to analysis.

7.0 HEALTH AND SAFETY

7.1 The toxicity or carcinogenicity of standards and reagents present by themselves and/or in complex mixtures may not have been established. Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes. Use fume hood, gloves and laboratory coats when performing analysis.

7.2 Since samples may contain high levels of other explosive compounds in addition to nitrocellulose, the target analyte for this procedure, soil samples should not normally be dried in an oven at temperatures greater than room temperature. Contact supervisor, Operations Manager or the Project Manager for project specific requirements. Handle samples with caution when preparing and analyzing sample extracts. Samples containing greater than 2 percent TNT must be handled with extra caution, by not grinding these samples and using very small aliquots of the sample. Since lumps of dry chemicals could be raw explosives, contact laboratory manager prior to handling.

7.3 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 Acetone, the extraction solvent used in this procedure is extremely flammable. All procedures involving the evaporation and/or transfer of acetone solutions should be performed in a hood.

7.5 Acids (sulfuric acid) and alkali (sodium hydroxide) used in this procedure are highly corrosive. Gloves and safety glasses should be worn to prevent contact with skin and eyes when handling these reagents.

7.6 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

7.7 Nitrocellulose standard is shipped moistened with isopropanol to minimize the chance of combustion. Store this standard in airtight containers to minimize evaporation of isopropanol from this reagent.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Soil samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters after the solvent(s) have been allowed to evaporate in a hood.

8.4 Return soil samples containing explosives to the client for proper disposal.

9.0 SAMPLE EXTRACTION PROCEDURE

Document sample weights, volumes, extraction QC batch number, lot number for the reagents, extract volumes, date, analyst initials, etc. in the sample preparation log books (see Appendix 4 for details). Section 9.1 describes the triple aqueous wash procedure that is employed to remove inorganic nitrates and nitrites. Section 9.2 describes the organic extraction procedure to extract nitrocellulose from the soil samples. Section 9.3 describes alkaline hydrolysis of the NC residues to inorganic nitrates. Section 9.4 describes the cadmium reduction procedure for converting inorganic nitrate to nitrite. Use air-dried soil samples for the following extraction procedure.

9.1 Aqueous pre-wash to remove inorganic nitrates/nitrites:

9.1.1 Accurately weigh 10-g of air-dried sample into a 40-ml VOA vial. Weigh two additional aliquots of the sample selected for matrix spike (MS) and matrix spike duplicate (MSD) analyses. Weigh 10-g of clean laboratory sand (Section 5.1.7) for method blank (MB), laboratory control sample (LCS), and LCS duplicate (LCSD).

9.1.2 Prepare LCS, LCSD, MS, and MSD samples by pipetting 1-ml of the NC stock standard solution in acetone into the respective 40-ml VOA vials. Do not spike MB or the environmental samples. Allow the acetone to evaporate in a hood (overnight, if necessary) prior to continuing with the analysis. NOTE: The acetone must be completely evaporated off in order to obtain reproducible recoveries.

9.1.3 Add 20-ml of Type I reagent water to each vial. Vortex each vial for a minute and place the vials on a mechanical shaker at maximum speeds for 15-minutes.

9.1.4 Remove the vials from the shaker and place them in a centrifuge at 2000- to 2500- rpm for 15-minutes. Pad the vials to prevent breakage.

9.1.5 Decant the water from the vials, taking care to not include soil fines. Use Pasteur pipettes to carefully remove water, if necessary.

9.1.6 Repeat steps 9.1.3 to 9.1.5 two more times. Vigorous shaking and mixing on a vortex mixer may be necessary to dislodge soil from the bottom of the vials after centrifuging operations. The samples are now ready for organic extraction.

9.2 Organic extraction to recover NC:

9.2.1 Add 20-ml of acetone to the soil in 40-ml VOA vials prepared as described in Section 9.1. Cap and shake (vortex mixer) the vial to dislodge soil from the bottom of the vial.

9.2.2 Place the vials in a water-cooled ultrasonic bath for 10- to 12-hours (overnight, if necessary). Keep cold water running to keep the sample cool during this operation.

9.2.3 Remove the vials from the ultrasonic bath, wipe the outside of the vials with paper towels and place them in a centrifuge at 2000- to 2500- rpm for 15-minutes. Pad the vials to prevent breakage.

9.2.4 Decant the acetone layer from the vials into a second-set of properly labeled, 40-ml vials, taking care to not include soil fines. Use Pasteur pipettes to carefully recover acetone extract, if necessary.

9.2.5 Add 5-ml of acetone to the vials containing the soil. Cap and shake (vortex mixer) the vial to dislodge soil from the bottom of the vials and to re-suspend them in acetone.

9.2.6 Place the vials in a centrifuge at 2000- to 2500- rpm for 15-minutes. Pad the vials to prevent breakage.

9.2.7 Decant the acetone extract into the vials containing the original acetone extract, taking care to not include soil fines. Use Pasteur pipettes to carefully recover acetone extract, if necessary.

9.2.8 Repeat steps 9.2.5 to 9.2.7 one more time. The combined acetone extracts (approximately 30-ml in volume) is now ready for the next step, which involves conversion of the organic nitrate to inorganic nitrate.

9.3 Conversion of organic nitrate to inorganic nitrate:

9.3.1 Evaporate the acetone extracts to dryness in a warm water bath at <50°C. This operation must be performed in a hood.

9.3.2 Add 5-ml of 1N NaOH (Section 5.1.8) solution to the residue in each 40-ml vial.

9.3.3 Cap the vial tightly and place in a 95°C water bath for 30-minutes. The temperature should not exceed 100°C to avoid significant pressure build-up.

9.3.4 Cool the vials to room temperature. Add 5-ml of Type I water and swirl to mix. Add 5-ml of 1N sulfuric acid (Section 5.1.9) and swirl to mix.

9.3.5 Quantitatively transfer the solution to appropriately labeled, clean 25-ml volumetric flasks, using Type I water to rinse the vials. Dilute to volume (25-ml) with Type I water.

9.3.6 The organic nitrates are now converted into inorganic nitrates. Since the aqueous digestates contain significant amount of sulfates (approx. 0.2 N), they may not be suitable for nitrate analysis using ion chromatography (EPA Method 300.0 or SW-846 Method 9056). Therefore, the nitrate determination is performed using spectrophotometric procedures after cadmium reduction of the nitrates to nitrite.

9.4 Cadmium reduction of inorganic nitrate: If the digestates (Section 9.3.5) are turbid, they may be filtered through 0.45-µ filter prior to cadmium reduction. If filtration does not solve the turbidity problem, add 0.25-ml zinc sulfate solution (Section 5.2.17) to 25-ml of the sample digestate and mix thoroughly. Add 0.1 to 0.125-ml of sodium hydroxide solution (Section 5.2.18) to obtain a pH of 10.5 as determined with a pH meter. Let the treated sample stand a few minutes to allow the heavy flocculent precipitate to settle. Clarify by filtering through a glass fiber filter or a 0.45-µ membrane filter. If oil and grease are present, they may be removed by adjusting the pH of 25-ml of the filtered sample to 2 by addition of conc. HCl (Section 5.2.4), and extracting the oil and grease from the aqueous digestates with two 25-ml portions of hexane (Section 5.2.21).

9.4.1 If the pH of the aqueous digestate (Section 9.3.5 or 9.4) is below 5 or above 9, adjust to between 5 and 9 with either conc. HCl or conc. HN_4OH. This is done to insure a sample pH of 8.5 after step 9.4.2.

9.4.2 To 25-ml of the sample digestate or an aliquot diluted to 25-ml, add 75-ml of ammonium chloride-EDTA solution (Section 5.2.14) and mix.

9.4.3 Pour sample into the cadmium reduction column (Section 5.2.20) and collect sample at a rate of 7- to 10-ml per minute. Discard the first 25-ml, collect the rest of the reduced sample (approximately 70-mL) in an appropriately labeled, clean, 100-ml plastic container.

9.4.4 Perform steps outlined in Sections 9.4.1 to 9.4.4 on all the calibration standards and the calibration blank (Sections 5.4.1 to 5.2.6).

9.4.5 Perform steps outlined in Sections 9.4.1 to 9.4.4 on quality control samples (MB, LCS, LCSD, MS, and MSD) and the associated environmental samples.

9.4.6 Reduced samples should not be allowed to stand longer than 15-minutes before the addition of color reagent and spectrophotometric analysis as described in Section 10.

10.0 SPECTROPHOTOMETRIC ANALYSIS

10.1 Set up the spectrophotometer in accordance with the manufacturer instructions.
10.2 Prepare a color reagent blank by adding 0.2-ml of the color reagent (Section 5.2.16) to 5-ml of reagent water (Section 5.1.1). Zero the instrument at 540-nm using this color reagent blank.
10.3 Perform color reaction on sample digestates (Section 9.4.1), calibration standard digestates (Section 9.4.2), and quality control sample digestates (Section 9.4.3) by adding 0.2-ml of the color reagent (Section 5.2.16) to 5-ml of the sample digestates (Section 9.4.1 to 9.4.3). See note (Section 9.4.4). Allow 10 minutes for color development.
10.4 Measure the absorbance at 540-nm within 2-hours of the color reaction and record them in instrument log books (See Appendix 4 for details).
10.5 If the concentration of sample exceeds 1.0-mg/L of NO_3-N, the remainder of the reduced sample may be used to make an appropriate dilution before performing the color reaction.
10.6 At least one nitrite standard should be compared to a reduced nitrate standard at the same concentration to verify the efficiency of the reduction column.
10.7 Calculate NO_3-N concentrations in sample digestates using the calibration curve and convert them to NC calculations in the soil sample as described in the following section.

11.0 CALCULATIONS

11.1 Plot the absorbance (y-axis) of the calibration blank and calibration standards (x-axis) from data collected as described in Section 10.3. Use linear regression to determine the slope, y-intercept and the correlation coefficient. The correlation coefficient should be greater than 0.995 for the calibration curve to be acceptable. Use an Excel spreadsheet (see Appendix 2 for details) to perform these calculations and to present data in graphical form.

11.2 Calculate the concentration of NO_3-N in sample digestates using the spreadsheet.

11.3 Calculate the nitrocellulose (NC) concentrations (mg/kg) from the volume (V) of the sample digestates (typically 25-ml), dilution factors employed (D) for the color reaction, dry weight (typically 10-g) of the sample (W) in grams, and the conversion factor (0.12). Do not use correction factors from the percent solids determination since air-dried samples are used for the analysis. The nitrocellulose (NC) standard used contains 12% of

NO_3-N by weight. Therefore, 0.12-mg of NO_3-N is equivalent to 1-mg of NC. Use the following equation for calculations:

$$\text{Nitrocellulose (mg/kg)} = \frac{(NO_3\text{-N in mg/L}) * (\text{V in ml}) * (0.001\ \text{L/ml}) * D}{(\text{W in g}) * (0.001\ \text{kg/g}) * 0.12}$$

11.4 Calculate percent recoveries for LCS, LCSD, MS, and MSD from the amount spiked (typically 100-mg/kg) and the amount present in the sample using the spreadsheet.

11.5 Calculate practical or method quantitation limit (PQL or MQL) for the analysis using the equation described in section 11.3 using the either the lowest or the second lowest point on the calibration curve (typically 0.05-mg/L NO_3-N). The PQL for a typical scenario (10-g sample, 25-ml digestates, D=1) calculated using the lowest point on the calibration curve is 1.04-mg/kg. The MQL used by the laboratory for NC analysis in soil samples is 2-mg/kg.

11.6 The upper limit of calibration, which corresponds to 1-mg/L NO_3-N is 20.8-mg/kg NC. Therefore, samples that contain greater than 20.8-mg/kg NC (e.g. LCS and MS spikes, which are typically 100-mg/kg per USACE protocol) will require dilution (typically 1:10) prior to the color reaction. The upper limit of the calibration can be extended through appropriate dilution of the sample digestates either before or after the cadmium reduction step but prior to the color reaction step.

11.7 The method detection limit (MDL) for this procedure is calculated from the MDL for the spectrophotometric step (approximately 0.01-mg/L) using the equation described in section 11.3. Using this procedure, the estimated MDL for NC in soil is 0.2-mg/kg. See Table 1 for the results of MDL studies in soil (also Appendix 7 for details). The reporting limit typically used by the laboratory for NC analysis is 1-mg/kg to reflect problems associated with variability in sample preparation procedures.

12.0 DATA VERIFICATION AND VALIDATION - This section will provide guidance when evaluating and validating data generated by this SOP. It provides procedures to assess if data produced is valid and legally defensible. Document review and comments on a checklist (see Appendix 3 for details). Verify that sample preparation logs (see Appendix 4 for details) are complete and instrument run logs are available for review. Anomalies and exceptions should be documented in the checklist. Attach sample result forms, QC summary, etc. as shown on the checklist. For example, samples run under rapid turnaround conditions may require the reporting of results that may not meet all method requirements. Such exceptions and data qualifications necessary to meet rapid TAT conditions will be documented on these checklists.

12.1 HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

12.1.1 SOIL – A 14 day holding is used for soil and waste samples.

12.1.2 TCLP – TCLP leachates must be extracted within 7 days after the TCLP extraction.

12.1.3 Holding times are established by comparing the chain of custody sampling date with the date of the injection after purging. TCLP holding times must be established by comparing first the COC to the leaching date and then the leaching date to the injection date. See Table III for applicable holding times.

12.1.4 ACTION – Since this is an unusual test, holding times may not have been established. If holding times used by the laboratory are exceeded, contact the client. All positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or invalid (I).

12.2 INITIAL CALIBRATION – Instrument calibration criteria are established to ensure that the calibration procedure used for the determination of nitrate in sample digestates is capable of producing acceptable quantitative data. The calibration curve or initial calibration (ICAL) demonstrates that the cadmium reduction and spectrophotometric procedures are capable of producing data within a linear range. Document the calibration curve in Form-6 equivalents or in linear regression graphs (see Appendix 2 for details).

12.2.1 Check to see that the correlation coefficient for linear regression is 0.995 or higher.

12.2.2 ACTION – A new standard curve should be prepared prior to sample cadmium reduction and spectrophotometric analysis. Sample digestion (Section 9) need not be repeated.

12.3 INITIAL (ICV) AND CONTINUING (CCV) CALIBRATION VERIFICATION – The calibration verification is used to check that the ICAL is still valid. Analyze one ICV before the analysis of any sample digestates and one CCV for every 10 samples thereafter. The ICVs and CCVs go through cadmium reduction and color reaction steps. Document calibration verification data in Excel spreadsheet printouts.

12.3.1 Check to see if the %D for ICVs and CCVs are less than 20%.

12.3.2 ACTION – If the %D is >20%, a new standard curve and additional calibration verifications (ICV and CCVs) should be prepared and the analysis repeated. Sample digestion (Section 9) need not be repeated. For time critical projects data not meeting ICV/CCV criteria may be qualified as estimated (J) by the user (client). Report non-detects without qualifiers and mention these outliners in the case narrative.

12.4 METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Document MB results in Form-1 equivalents (see Appendix 8 for details). Document association of project samples and QC samples with the MB in Form-4 equivalents (QC association forms, see Appendix 4 for details). Sample data qualification to alert blank contamination is performed by the laboratory. Sample data qualification based on all the blank results (laboratory MB, equipment blank, and other field blanks) is performed by the user (client). The processing of blanks helps eliminate reporting of false positives in project reports.

12.4.1 Method blanks should be free of all target analytes and other interfering non-target analytes. To be acceptable, blank results should not exceed 1-mg/kg (one half of the applicable MQL) for NC in soil.

12.4.2 Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples. If levels greater than one half the MQLs are present, inspect and correct the problem. Reanalyze all associated samples. Contact client if other criteria are applicable. The exception may be quick turn around projects and projects with blank criteria.

12.4.3 ACTION – If the blank amounts are less than the reporting level (1-mg/kg) for NC in soil, no action is taken.

12.4.4 ACTION - If the blank amounts are greater than one half of the MQL, all positive results are reported with a "B" code.

12.4.5 ACTION – If the analyte is detected only in the blank and not in any of the associated samples, then no action is taken.

12.4.6 ACTION – Samples that contain the analyte at levels greater than 10 times the blank level need not be re-extracted.

12.5 SURROGATE RECOVERIES – Surrogates are not employed for this analysis.

12.6LABORATORY CONTROL STANDARD (LCS) – A LCS analysis is used to evaluate method performance in the absence of matrix effects. Whenever possible, the LCS should be from a second source standard. For this analysis, calibration is performed using nitrate standards, and spiked sample analyses (LC, MS, etc.) are performed using NC solutions for spiking. Report LCS results in Form-1 equivalents and LCS recoveries and RPD for LCS duplicates in Form-3 equivalents (see Appendix 5 for details).

12.6.1 The QC limits for LCS recoveries are shown in Table 1.

12.6.2 Check the LCS recoveries and the RPD for duplicate analyses are within the acceptable QC limits. Flag outliers with an asterisk (*).

12.6.3 ACTION – The method used for NC has not been well tested to establish performance requirements. Therefore, no action is taken on LCS/LCSD recovery data alone either to reanalyze samples and/or to qualify associated samples for some projects. If recoveries are outside QC limits, reanalysis of the LCS and associated samples may be necessary depending on client and project requirements. Check with the project manager about project specific requirements. Professional judgment may be used by the user in evaluating sample data qualification options in conjunction with other QC data for the project.

12.7MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike data are used to evaluate the precision and accuracy of the analytical method on project samples. Document MS results in Form-1 equivalents and recoveries/RPDs in Form-3 equivalents (see Appendix 6 for details). Document

12.7.1 QC limits for MS/MSD percent recoveries and the relative percent difference (RPD) between the MS and MSD results are shown in Table 1.

12.7.2 Check the results for the MS/MSD recoveries are within the QC limits (50-150). Flag outliers with an asterisk (*).

12.7.3 Perform one manual calculation using raw data and extraction sheet information.

12.7.4 Calculate the relative percent difference (RPD) between MS and MSD recoveries. If the RPD is greater than the administrative QC limit (50%), flag it with an asterisk (*).

12.7.5 ACTION – The method used for NC has not been well tested to establish performance requirements. Therefore, no action is taken on MS/MSD recovery data alone. Since matrix interference, high levels of NC in the sample selected for MS analysis, etc. could lead to lead to serious analytical problems, the supervisor should be consulted when significant outliers are noted. Document QC outliers for %recovery and RPD on the checklist and on the case narrative for client reports. No action may be taken on MS/MSD recovery data alone either to reanalyze samples and/or to qualify associated samples either by the analyst or by the end user. Professional judgment may be used by the user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

13.0 REFERENCES

13.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8330, Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC), September 1994.

13.2 USATHAMA Method LF03, Determination of Nitrocellulose in Soil by Autoanalyzer.

13.3 Missouri River Division (MRD) laboratory standard operating procedure, Determination of nitrocellulose in soil, received September 2000.

13.4 USEPA Manual of methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Nitrogen, Nitrate-Nitrite, Method 353.3, Spectrophotometric, manual cadmium reduction.

13.5 USEPA Manual of methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Nitrogen, Nitrite, Method 354.1, Spectrophotometric.

13.6 AML Standard Operating Procedures, Q-Water-RC.

13.7 AML Standard Operating Procedures, Q-WasteMgmt-RD.

13.8 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

13.9 AML Standard Operating Procedures, Q-ControlCharts-RC.

Table 1
Soil MDLs, MQLs, and QC Limits for LCS/LCSD and MS/MSD

Nitrocellulose Analysis by Spectrophotometry														
Analytical Management Laboratories														
Matrix: Soil	**Sample Preparation**				**Sample Analysis**						**Technique**			
Parameter	Method No.		Date		Method No.		Method No.		Date					
Nitrocellulose	USATHAMA LF03		4/19/01		EPA 353.3		EPA 354.1		4/20/01		Spectrophotometry			
	Spike	Mean	S.D.	**MDL**			**MQL**			**LCS Limits**		**MS Limits**		**RPD**
Parameter	Amount	mg/kg	mg/kg	**mg/kg**			**mg/kg**			Lower	Upper	Lower	Upper	Limit
	mg/kg									**%R**	**%R**	**%R**	**%R**	%
Nitrocellulose	2.0	2.14	0.12	0.37			2.0			60	120	40	140	50

Table 2
List of Appendices

Appendix Number	Appendix Topic
1	Figure 1 - Cadmium reduction Column (from EPA 353.3)
2	Excel Spreadsheets used for data processing – Printouts Initial Calibration (ICAL) Summary (Form-6 Equivalents) Calibration Verification (ICV and CCV) Summary (Form-7 Equivalents) Sample Results Summary (Benchsheets)
3	Checklist for Review and Data Verification
4	QC Association Form (Form-4 Equivalents) Nitrocellulose Sample Preparation Log Nitrate Analysis Logs
5	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
6	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
7	Method Detection Limit (MDL) Studies Summary
8	Sample Reports (Form-1 equivalents)

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. I-Nitrocellulose-Water-RC

NITROCELLULOSE IN WATER

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) is based on the method developed by USACE CRREL Laboratory. Samples need complicated digestion, extraction, and cleanup procedures prior to instrumental analysis. Instrumental analysis can be performed by ion chromatography or by spectrophotometry as described in the AML SOP on the determination of nitrocelulose (NC) in soil.

1.2 All analysts performing this method should have 6-months prior experience in performing wet chemistry analysis including the analysis of nitrate and nitrite.

1.3 The sample extraction and cleanup procedures described in this SOP should be followed strictly as written, disregarding minor inconsistencies between NC spike levels for spiked sample (LCS and MS) analysis and the calibration curves for nitrate determination. Any modifications to this SOP should be reviewed and approved by the supervisor and QA Manager/Director prior to implementation and incorporation into the SOP.

1.4 The linear concentration range for NC is approximately 4- to 800-µg/L.

2.0 SUMMARY OF METHOD

2.1 The nitrocellulose (NC) is isolated from a water sample by vacuum filtration through a 0.02-micron inorganic (ANOPORE) filter. The membrane is washed with methanol to remove interfering nitrate esters such as PETN.

2.2 The filter membrane is then soaked in acetone and the acetone extract collected. The acetone extract is evaporated to dryness and the residue containing nitrocellulose is hydrolyzed with caustic soda to liberate nitrate and nitrite.

2.3 Nitrocellulose (NC) is determined based on the amount of nitrate and nitrite produced following hydrolysis of NC by sodium hydroxide.

2.4 When ion chromatography is used for the determination, the hydrolyzed sample is neutralized by passage through a cation exchange sample preparation cartridge.

2.5 When manual spectrophotometry is used for the determination, the caustic soda is neutralized with sulfuric acid and manual cadmium reduction (EPA Method 353.3) is employed on the aqueous digestates to convert the nitrate into nitrite. The total nitrite is determined manually using EPA Method 354.1 by spectrophotometry.

2.6 The procedures used for cadmium reduction and spectrophotometric determination of the inorganic nitrite/nitrate determination in aqueous samples are incorporated by reference to the AML SOP on the determination of nitrocellulose in soil.

2.7 The procedures used for the inorganic nitrite/nitrate determination in aqueous samples are incorporated by reference to the USACE CRREL SOP on the determination of nitrocellulose in water.

2.8 The NC determination employs total organic nitrate in the sample. Therefore, separate values for nitrate and nitrite in aqueous sample digestates, which are obtained by carrying out the spectrophotometric procedure first with, and then without, the Cu-Cd reduction step is not necessary.

3.0 INTERFERENCES

3.1 Aqueous samples may contain high levels of inorganic nitrate and nitrite, which are removed by aqueous extraction procedures described in this SOP. Since nitrocellulose is determined from the nitrate/nitrite content in organic solvent extracts, incomplete removal of inorganic nitrate prior to solvent extraction may result in positive bias for nitrocellulose.

3.2 Interferences may also be caused by other nitrate/nitrite containing organic compounds (e.g. explosives) that are insoluble in water and extractable by acetone. A methanol wash of the sample may be used to either reduce or to remove some of these organic interferences. However, methanol was procedure has not been evaluated for routine application.

3.3 Solvents, reagents, glassware can produce laboratory-generated artifacts, and background interferences, which can produce false positive results. A method blank (MB) analysis is used to verify the absence of these contaminants. If the MB contains the target analyte at or above one half of the practical or method quantitation limit (PQL or MQL), appropriate corrective action may be necessary.

3.4 Build up of suspended matter in the cadmium reduction column will restrict sample flow. Since nitrate-nitrogen is found in a soluble state, the sample may be pre-filtered through a glass fiber filter or a 0.45-μ membrane filter. Highly turbid samples may be pretreated with zinc sulfate before filtration to remove the bulk of particulate matter present in the sample.

3.5 Sample results exceeding site specific action level should be carefully evaluated to determine if potential interferences exist. Recommendations should be made to the Client Project Manager to perform additional analyses such as the analysis for nitroaromatic explosives (SW-846 Method 8330 and 8332) and inorganic nitrate and nitrite (EPA 300.0, and/or 353.3). The laboratory should not perform such additional analyses without prior approval by the Client Project Manager.

3.6 All glassware used during the extraction procedures is washed using the following procedure:

- 3.6.1 Soak glassware in hot soap and water
- 3.6.2 Scrub using a laboratory brush
- 3.6.3 Rinse with tap water
- 3.6.4 Rinse with deionized water 3 times
- 3.6.5 Rinse with acetone and dry
- 3.6.6 Store glassware in a clean area.

4.0 APPARATUS AND MATERIALS

- 4.1 Centrifuge, Fisher Scientific Model Marathon 3200. See instrument manual for instrument operating conditions.
- 4.2 Ultrasonic Bath, Branson Model 8210. Maintain at room temperature or less by cycling tap water through the bath during sample extraction. Consult with analysts experienced in explosive analysis in soil by SW-846 Method 8330 for detailed operation procedures. See instrument manual for instrument operating conditions.
- 4.3 Equipment (home-built) for air-drying soil samples for NC and explosives analysis.
- 4.4 VOA vials (40 ml) with Teflon lined caps.
- 4.5 Vortex Genie
- 4.6 Water Bath
- 4.7 Wrist Action Shaker

4.8 Cadmium Reduction Columns
4.9 Hamilton syringes- various sizes
4.10 Vacuum Desiccator
4.11 Volumetric flasks-Class A- various size
4.12 Air drying oven
4.13 Spectrophotometer, Milton Roy, Model Spectronic 20. See instrument manual for instrument operating conditions. Use at 540-nm, providing a light path of 1-cm or longer.
4.14 Cadmium reduction column: The column (see Appendix 1 for details) was constructed from a 100-ml pipette by removing the top portion. This column may also be constructed from two pieces of tubing joined end to end. A 10-mm length of 3-cm I.D. tubing is joined to a 25-cm length of 3.5-mm I.D. tubing.

5.0 REAGENTS

All chemicals used are ACS Reagent grade or better. If contamination is found at or above method detection limit, all suspected chemicals are screened individually and replaced.

5.1 The following reagents are used with the NC determination part of the method:
- 5.1.1 Reagent water: Filtered (0.45 μ), Type I water from USFilter water system.
- 5.1.2 Acetone - Pesticide or HPLC grade (EM Science or equivalent). Acetone is extremely flammable. All procedures involving transfer of acetone solutions should be performed in a hood.
- 5.1.3 Methanol – Pesticide or HPLC grade (EM Science or equivalent). Methanol is extremely flammable. All procedures involving transfer of acetone solutions should be performed in a hood.
- 5.1.4 Sodium Hydroxide, Fisher Scientific, ACS Reagent grade. Sodium hydroxide is corrosive.
- 5.1.5 Sulfuric Acid, concentrated, Fisher Scientific, ACS Reagent grade. Acids are corrosive.
- 5.1.6 Nitrocellulose, Aldrich cat # 43503-1 containing 12% nitrogen. Nitrocellulose (NC) is shipped with isopropanol to minimize the chance of combustion. Prior to preparing stock solutions, prepare this reagent by spreading approximately 1-g evenly over the bottom of a weighing boat. Place the weighing boat in a desiccator for several days to eliminate the isopropanol and to dry the nitrocellulose. Use the dried nitrocellulose to prepare stock standards.
- 5.1.7 Laboratory sand. Pre-wash with Type II water, followed by Type I water, dry in oven and store in an appropriately labeled, clean, glass container.
- 5.1.8 Sodium hydroxide (1N) solution - Dissolve 40-g of NaOH in 1000-ml of Type I or II water, mix well, and transfer to a properly labeled plastic container.
- 5.1.9 Sulfuric acid (1N) solution - Add 28-ml of concentrated H_2SO_4 to 500-ml of Type I or II reagent water in a 1000-ml volumetric. Dilute to volume with reagent water, mix well, and transfer to a properly labeled plastic container.

5.2 The following reagents are used with the cadmium reduction and nitrite determination part of the method:
- 5.2.1 Granular cadmium, 40-60 mesh, Fisher Scientific, ACS reagent grade.
- 5.2.2 Copper sulfate ($CuSO_4.5H_2O$), Fisher Scientific, ACS reagent grade.

5.2.3 Potassium nitrate, Fisher Scientific, ACS reagent grade.
5.2.4 Hydrochloric acid, concentrated, Fisher Scientific, ACS reagent grade.
5.2.5 Phosphoric acid, concentrated, Fisher Scientific, ACS reagent grade.
5.2.6 Sulfanilamide, Fisher Scientific, ACS reagent grade.
5.2.7 N(1-napthyl)-ethylenediamine dihydrochloride, Fisher Scientific, ACS reagent grade.
5.2.8 Ammonium chloride, Fisher Scientific, ACS reagent grade.
5.2.9 Disodium ethylenediamine tetracetate (EDTA), Fisher Scientific, ACS reagent grade.
5.2.10 Zinc Sulfate ($ZnSO_4.7H_2O$), $CuSO_4.5H_2O$, Fisher Scientific, ACS reagent grade.
5.2.11 Ammonium Hydroxide, concentrated, Fisher Scientific, ACS reagent grade.
5.2.12 Dilute hydrochloric acid, 6N: Dilute 50-ml of conc. HCl to 100-ml with Type I or II reagent water.
5.2.13 Copper Sulfate Solution, 2% - Dissolve 20-g of $CuSO_4 \bullet 5H_2O$ in 500-ml of reagent water and dilute to 1-liter.
5.2.14 Ammonium chloride - EDTA solution: Dissolve 13-g ammonium chloride and 1.7-g of EDTA in 900-ml of reagent water. Adjust the pH to 8.5 with conc. ammonium hydroxide and dilute to 1-liter with reagent water.
5.2.15 Dilute ammonium chloride-EDTA solution: Dilute 300-ml of ammonium chloride-EDTA solution from the previous step to 500- ml with distilled water.
5.2.16 Color reagent: Dissolve 10-g sulfanilamide and 1-g N(1-naphthyl)-ethylene-diamine dihydrochloride in a mixture of 100-mL conc. phosphoric acid and 800-ml reagent water. Dilute to 1-liter with reagent water.
5.2.17 Zinc sulfate solution: Dissolve 100 g $ZnSO_4 \bullet 7H_2O$ in distilled water and dilute to 1 Liter.
5.2.18 Sodium Hydroxide Solution, 6N: Dissolve 240-g NaOH in 500- ml reagent water, cool and dilute to 1-liter.
5.2.19 Copper-Cadmium Reagent: The cadmium granules (new or used) are cleaned with dilute HCL and copperized with 2% solution of copper sulfate in the following manner: Wash the cadmium with dilute HCL (Section 5.2.12) and rinse with reagent water. The color of the cadmium should be silver. Swirl 25-g cadmium (Section 5.2.1) in 100-ml portions of a 2% solution of copper sulfate (Section 5.2.13) for 5-minutes or until blue color partially fades, decant and repeat with fresh copper sulfate until a brown colloidal precipitate forms. Wash the copper-cadmium with reagent at least 10-times to remove all the precipitated copper. The color of the cadmium so treated should be black.
5.2.20 Copper-Cadmium Reduction Column: Insert a glass wool plug into the bottom of the reduction column and fill with reagent water. Add sufficient copper-cadmium granules to produce a column 18.5-cm in length. Maintain a level of distilled water above the copper-cadmium granules to eliminate entrapment of air. Wash the column with 200-ml of dilute ammonium chloride-EDTA solution (Section 5.2.15). The column is then activated by passing through the column 100-ml of a solution composed of 25-ml of a 1.0-mg/L NO_3-N standard and 75-ml of ammonium chloride-EDTA solution (Section 5.2.14). Use a flow rate between 7- and 10-ml per minute.

5.2.21 Hexane - ACS Reagent grade (EM Science or equivalent). Hexane is extremely flammable. All procedures involving transfer of hexane solutions should be performed in a hood.

5.3 Stock Standard Solutions. Prepare stock solutions of nitrocellulose, inorganic nitrate and inorganic nitrite to be used in various steps.

5.3.1 Nitrocellulose (NC) Stock Standard Solution (1000-ug/ml in acetone). Weigh 0.100-g of the dried nitrocellulose (See 5.1.4) into a 100-ml volumetric. Add approximately 50-ml of acetone to dissolve the NC. Dilute to volume with acetone and mix well. Transfer to a properly labeled 125-ml glass bottle and store at 4°C . The stock standard solution expires in 1-year from the date of preparation. Replace sooner if signs of degradation are observed.

5.3.2 Stock Nitrate Solution: Dissolve 7.218-g KNO_3 (Section 5.2.3) in reagent water and dilute to 1000-mL. Preserve with 2-ml of chloroform per liter. This solution (1000-mg/L NO_3-N) is stable for at least 6-months. 1.0-mL = 1.00-mg NO_3-N.

5.3.3 Standard Nitrate Solution: Dilute 10.0-ml of nitrate stock solution (Section 5.3.2) to 1000-ml with reagent water. 1.0-ml = 0.01-mg NO_3-N. Prepare this solution (10-mg/L NO_3-N) fresh daily.

5.3.4 Purchase a prepared, certified 1000-mg/L standard Nitrate solution from suitable vendors (e.g. Ultra Scientific)

5.3.5 Purchase a prepared, certified 1000-mg/L standard Nitrite solution (e.g. Ultra Scientific).

5.4 Nitrate Calibration Standards - These are prepared fresh daily from the 10-mg/L NO_3-N standard nitrate solution (Section 5.3.3). Six solutions consisting of one calibration blank and five nitrate concentrations are prepared by diluting the standard nitrate solution with reagent water (Section 5.1.1).

5.4.1 Concentration (mg/L) of NO_3-N = 0.00: Dilute 0.00-ml of the standard nitrate solution to 100-ml with reagent water.

5.4.2 Concentration (mg/L) of NO_3-N = 0.05: Dilute 0.50-ml of the standard nitrate solution to 100-ml with reagent water.

5.4.3 Concentration (mg/L) of NO_3-N = 0.10: Dilute 1.00-ml of the standard nitrate solution to 100-ml with reagent water.

5.4.4 Concentration (mg/L) of NO_3-N = 0.20: Dilute 2.00-ml of the standard nitrate solution to 100-ml with reagent water.

5.4.5 Concentration (mg/L) of NO_3-N = 0.50: Dilute 5.00-ml of the standard nitrate solution to 100-ml with reagent water.

5.4.6 Concentration (mg/L) of NO_3-N = 1.00: Dilute 10.0-ml of the standard nitrate solution to 100-ml with reagent water.

5.5 See Appendix for a

5.6

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 One liter glass jars with Teflon lined screw caps may be used for collecting water samples. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Quality control samples such as MS and MSD should

be treated as regular environmental samples. Upon receipt and log-in, these sample containers should be stored in the walk-in cooler.

6.2 Samples must be extracted and analyzed within the specified holding times for the results to be considered reflective of total concentrations. Holding times may not have been established for this unusual parameter. Due to the high stability of the nitrocellulose, the holding time concept may not even be applicable. Analytical data generated outside of the specified holding times, if any should be considered to be minimum values only. The laboratory holding time for the extraction of water sample is 28 days from the date of sample collection or receipt. Samples extracts are stored in amber bottles and/or vials at 4°C just prior to analysis.

7.0 HEALTH AND SAFETY

7.1 The toxicity or carcinogenicity of standards and reagents present by themselves and/or in complex mixtures may not have been established. Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes. Use fume hood, gloves and laboratory coats when performing analysis.

7.2 Since samples may contain high levels of other explosive compounds in addition to nitrocellulose, the target analyte for this procedure, soil samples should not normally be dried in an oven at temperatures greater than room temperature. Contact supervisor, Operations Manager or the Project Manager for project specific requirements. Handle samples with caution when preparing and analyzing sample extracts. Samples containing greater than 2 percent TNT must be handled with extra caution, by not grinding these samples and using very small aliquots of the sample. Since lumps of dry chemicals could be raw explosives, contact laboratory manager prior to handling.

7.3 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 Acetone, the extraction solvent used in this procedure is extremely flammable. All procedures involving the evaporation and/or transfer of acetone solutions should be performed in a hood.

7.5 Acids (sulfuric acid) and alkali (sodium hydroxide) used in this procedure are highly corrosive. Gloves and safety glasses should be worn to prevent contact with skin and eyes when handling these reagents.

7.6 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

7.7 Nitrocellulose standard is shipped moistened with isopropanol to minimize the chance of combustion. Store this standard in airtight containers to minimize evaporation of isopropanol from this reagent.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

8.3 Soil samples that have undergone extraction should be free of contaminants. They can be disposed off into the laboratory sink and/or sanitary dumpsters after the solvent(s) have been allowed to evaporate in a hood.
8.4 Return soil samples containing explosives to the client for proper disposal.

9.0 SAMPLE EXTRACTION PROCEDURE

9.1 See Appendix 1 for this procedure.

10.0 SAMPLE ANALYSIS

10.1 See Appendix 1 for analysis by ion chromatography (IC). See also AML SOP on the analysis of anions by IC for other details.
10.2 See AML SOP on NC in Soil for analysis by spectrophotometry.

11.0 CALCULATIONS

11.1 See Appendix 1 for analysis by ion chromatography (IC). See also AML SOP on the analysis of anions by IC for other details.
11.2 See AML SOP on NC in Soil for analysis by spectrophotometry.

12.0 DATA VERIFICATION AND VALIDATION -

12.1 See referenced AML SOP on nitrocellulose analysis in soil for details.

13.0 REFERENCES

13.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8330, Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC), September 1994.
13.2 US Army Engineer Research and Development Center, USA CRREL, Analytical Method for the Determination of Nitrocellulose in Water, 1989.
13.3 Missouri River Division (MRD) laboratory standard operating procedure, Determination of nitrocellulose in soil, September 2000.
13.4 USEPA Manual of methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Nitrogen, Nitrate-Nitrite, Method 353.3, Spectrophotometric, manual cadmium reduction.
13.5 USEPA Manual of methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Nitrogen, Nitrite, Method 354.1, Spectrophotometric.
13.6 AML Standard Operating Procedure, I-Nitrocellulose-Soil-RB.
13.7 AML Standard Operating Procedures, Q-Water-RC.
13.8 AML Standard Operating Procedures, Q-WasteMgmt-RD.
13.9 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
13.10 AML Standard Operating Procedures, Q-ControlCharts-RC.

Table 1
Water MDLs, MQLs, and QC Limits for LCS/LCSD and MS/MSD

Nitrocellulose Analysis by Spectrophotometry														
Analytical Management Laboratories														
Matrix: Water	**Sample Preparation**				**Sample Analysis**						**Technique**			
Parameter	Method No.		Date		Method No.		Method No.		Date					
Nitrocellulose	USACE CRREL				EPA 353.3		EPA 354.1				Spectrophotometry			
	Spike	Mean	S.D.	**MDL**			**MQL**			**LCS Limits**		**MS Limits**		**RPD**
Parameter	Amount	Mg/L	Mg/L	**mg/L**			**mg/L**			Lower	Upper	Lower	Upper	Limit
	mg/L									**%R**	**%R**	**%R**	**%R**	%
Nitrocellulose	2.0			0.37			2.0			60	120	40	140	50

Table 2
List of Appendices

Appendix Number	Appendix Topic
1	US Army Corps of Engineers CRREL Analytical Method for the Determination of Nitrocellulose in Water
2	Figure 1 – Cadmium reduction Column (from EPA 353.3)
3	Excel Spreadsheets used for data processing – Printouts Initial Calibration (ICAL) Summary (Form-6 Equivalents) Calibration Verification (ICV and CCV) Summary (Form-7 Equivalents) Sample Results Summary (Benchsheets)
4	Checklist for Review and Data Verification
5	QC Association Form (Form-4 Equivalents) Nitrocellulose Sample Preparation Log Nitrate Analysis Logs
6	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
7	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
8	Method Detection Limit (MDL) Studies Summary
9	Sample Reports (Form-1 equivalents)

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. I-Oil&Grease-TPH-1664-RD

Oil & Grease or Hexane Extractable Materials (HEM) Total Petroleum Hydrocarbons or Silica Gel Treated HEM (SGT-HEM) by Gravimetric Methods (EPA 1664, 413.1, and 418.1, SM 5520, SW-846 9070A and 9071B)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Oil & Grease or Hexane Extractable Materials (HEM) Total Petroleum Hydrocarbons or Silica Gel Treated HEM (SGT-HEM) by Gravimetric Methods (EPA 1664, 413.1, and 418.1, SM 5520, SW-846 9070 and 9071A)

1.0 Scope and Application

1.1 This standard operating procedure (SOP) is based on EPA Method 1664, which replaced EPA 418.1, which employed Freon for sample extraction. This SOP is also based on related procedures such as EPA 413.1, Standard methods (5520-A, 5520-B, 5520-E, and 5520-F) and SW-846 Methods 9070 and 9071A.

1.2 This SOP is used to determine Oil and Grease (O&G) and Total Petroleum Hydrocarbons (TPH) by extraction with n-hexane and gravimetry with silica gel treatment (SGT). The n-hexane extractable material (HEM) is O&G and the HEM that is not adsorbed by silica gel (SGT-HEM) is TPH.

1.3 This method is for determination of HEM and SGT-HEM in surface and saline waters and industrial and domestic aqueous wastes. Extractable materials that may be determined are relatively non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases and related materials.

1.4 This method is used in the EPA's survey and monitoring programs under the Clean Water Act. "Oil and grease" is a conventional pollutant defined in the Act and codified at 40 CFR 401.16. The term hexane-extractable material" reflects that this method can be applied to materials other than oils and greases. Similarly, the term "silica gel treated hexane extractable material" reflects that this method can be applied to materials other than aliphatic petroleum hydrocarbons that are not absorbed by silica gel.

1.5 This method is not applicable to measurement of materials that volatilize at temperatures below approximately 85°C. Petroleum fuels from gasoline through #2 fuel oil may be partially lost in the solvent removal operation.

1.6 Some crude oils and heavy fuels oils contain a significant percentage of materials that are not soluble in hexane. Accordingly, recoveries of these materials may be low.

1.7 This method is capable of measuring in the range of 5 to 1000 mg/L, and may be extended to higher levels by analysis of a smaller sample volume collected separately.

1.8 For this method, the MDL has been determined as 1.4 mg/L for HEM and 1.6 mg/L for SGT-HEM, and the minimum level has been set at 5.0 mg/L for HEM and SGT-HEM.

2.0 Summary of Method

2.1 A 1-Liter sample is acidified to pH <2 and serially extracted three times with hexane in a separatory funnel. The extract is dried over sodium sulfate.

2.2 The solvent is evaporated from the extract and the HEM is weighed. If the HEM is to be used for the determinations of SGT-HEM, the HEM is redissolved in hexane.

2.3 For SGT-HEM determination, an amount of silica gel proportionate to the amount of HEM is added to the solution containing the redissolved HEM to remove absorbable materials. The solution is filtered to remove the silica gel, the solvent is evaporated, and the SGT-HEM is weighed.

2.4 Quality is assured through calibration and testing of the extraction, concentration and gravimetric systems.

3.0 Sample Collection, handling and Preservation

3.1 One liter amber glass bottles with Teflon lined screw caps may be used for collecting aqueous samples for oil and grease analysis. Order pre-cleaned bottles directly from one of several vendors (ESS, Eagle-Picher, Fisher Scientific, etc.). Add 4-mL of 1:1 sulfuric acid to the sample containers. Provide 1 bottle for each water sample to be collected. An additional sample container may need to be provided for re-extraction and re-analysis for potential QC failure.

3.2 Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. The laboratory should provide two additional sets of containers for MS/MSD sample collection. See project specific field sampling plan (FSP) for sample collection and preservation instructions.

3.3 Collect approximately one liter of representative sample in a glass bottle following conventional sampling practices, except that the bottle must not be pre-rinsed with sample before collection. If a sample is known or suspected to contain greater than 1000-mg/L of extractable material, collect a proportionately smaller volume of sample (the volume required will depend upon the estimated amount of extractable material) in a glass bottle. Collect an additional two aliquots of a sample for each set of ten samples or less for the matrix spike and matrix spike duplicate.

3.4 The high probability that extractable matter may adhere to sampling equipment and result in measurements that are biased low precludes the collection of composite sample for determination of oil and grease. Therefore, samples must be collected as grab samples. If a composite measurement is required, individual grab samples collected at prescribed time intervals must be analyzed separately and the concentrations averaged.

3.5 If samples are collected unpreserved and the analysis is to be delayed for more than four hours after sample collection, the pH of the sample should be adjusted to less than 2 with 1:1 sulfuric acid. Although the default amount for the preservative is 4-mL of 1:1 sulfuric acid per liter of sample collected, the actual volume of acid needed to adjust the pH to <2 should be determined in the field and modified as necessary to adjust the pH of samples collected to less than two.

3.6 Upon receipt and log-in, these sample containers should be stored in the walk-in cooler. All samples must be refrigerated at 0-4°C from the time of collection until extraction.

3.7 All samples must be analyzed within 28 days of the date and time of collection.

4.0 INTERFERENCES

4.1 Solvents, reagents, glassware and other sample-processing hardware may yield artifacts that affect results. Specific selection of reagents and purification of solvents may be required.

4.2 All materials used in the analysis shall be demonstrated to be free from interferences under the conditions of analysis by running laboratory blanks. Glassware is cleaned by washing in hot water containing detergent, rinsing with tap and distilled water, and rinsing with solvent or baking. Boiling flasks that will contain the extracted residue are dried in an oven at 105-115 °C and stored in a desiccator.

4.3 Sodium sulfate and silica gel fines have the potential to inflate results for HEM and SGT-HEM by passing through the filter paper. If the filter paper specified in this method is inadequate for the removal of these fines, use of a 0.45-micron filter is recommended.

4.4 Interferences extracted from samples will vary considerably from source to source, depending upon the diversity of the site being samples. For those instances in which samples are thought to consist of complex matrices containing substances (such as particulates or detergents) that may interfere with the extraction procedure, a smaller sample may need to be collected for analysis.

5.0 APPARATUS

5.1 Equipment for glassware cleaning

- 5.1.1 Laboratory sink.
- 5.1.2 Oven - Capable of maintaining a temperature within ± 5 °C in the range of 100-250°C.

5.2 Equipment for Calibration

- 5.2.1 Analytical Balance - Capable of weighing 0.1 mg.
- 5.2.2 Volumetric flask - Glass, 100-mL
- 5.2.3 Vials - Assorted sized, with PTFE-lined screw caps
- 5.2.4 Volumetric pipette - Glass, 5-mL

5.3 Equipment for sample extraction

- 5.3.1 Balance-Top loading, capable of weighing 500-2000 g within ± 1%
- 5.3.2 Glass stirring rod.
- 5.3.3 Separatory funnel - Glass, 2000-mL with PTFE stopcock.
- 5.3.4 Funnel - Large, glass, for pouring sample into separatory funnel

5.4 Equipment for removing water, sodium sulfate, and silica gel fines.

- 5.4.1 Funnel - Analytical, glass
- 5.4.2 Filter paper - Whatman No. 40 (or equivalent), to fit funnel.

5.5 Equipment for solvent evaporation

- 5.5.1 Water bath - capable of maintaining a temperature of at least 85 °C.
- 5.5.2 Flask, 125-mL
- 5.5.3 Tongs, for handling the flask
- 5.5.4 Desiccator - jar-type, capable of keeping the flask dry during cooling.

6.0 REAGENTS

6.1 Reagent water - Type II Deionized water

6.2 Hydrochloric or Sulfuric acid - ACS, 1:1. Mix equal volumes of concentrated HCl or H_2SO_4 and reagent water.

6.3 Hexane - 85% purity, 99.0% min. saturated C_6 isomers, residue less than 1 mg/L.

6.4 Acetone-ACS, residue less than 1 mg/L.

6.5 Sodium sulfate - ACS, granular anhydrous.

6.6 Boiling chips-silicon carbide or fluoropolymer.

6.7 Silica gel - Anhydrous, 75-150 micrometers. Dry at 200-250°C for 24 hours minimum and store in a desiccator or tightly sealed container. Determine the hexane soluble material content of the silica gel by extracting 30 g of silica gel with hexane and evaporating to dryness. The silica gel must contain less than 5 mg or hexane soluble material per 30 g (<0.17 mg/g).

6.8 Hexadecane-98% minimum purity.

6.9 Stearic acid - 98% minimum purity.

6.10 Hexadecane/stearic acid (1:1) spiking solution - Prepare in acetone at a concentration of 4 mg/mL each.

6.10.1 Place 400 ± 4-mg hexadecane and 400 ± 4-mg stearic acid in a 100-mL volumetric flask and fill to the mark with acetone.

6.10.2 After the hexadecane and stearic acid have dissolved, transfer the solution to a 100-150 mL vial with fluoropolymer-lined cap. Mark the solution level on the vial, and store the tightly capped vial in the dark at room temperature.

6.10.3 Immediately prior to use, verify the level on the vial and bring to volume with acetone, if required. Warm to re-dissolve all visible precipitate. *NOTE: If there is doubt of the concentration, remove 5.0 mL with a volumetric pipette, place in a tared weighing pan, and evaporate to dryness in a fume hood. The weight must be 40 ± 1 mg.*

6.11 Precision and recovery (PAR) standard - Spike 5.0 mL of the hexadecane/stearic acid spiking solution (6.10) into 950 mL of reagent water to produce concentration of approximately 20 mg/L each of hexadecane and stearic acid. The PAR standard is used for the determination of initial precision and recovery and ongoing precision and recovery. The laboratory control sample (LCS) is identical to the PAR standard. Vegetable oil used instead in conjunction with the older oil and grease method (EPA 413.1) should not be used instead of the hexadecane/stearic acid specified in Method 1664.

6.12 The spiking solution should be checked frequently for signs of degradation or evaporation using the test noted in section 6.10.3, and must be replaced after 6 months, or sooner if degradation has occurred.

7.0 QUALITY CONTROL

7.1 The minimum requirements of this method consist of analyzing a blank, LCS, and sample duplicate with each set of twenty samples.

7.2 An MS and MSD can also be performed if sufficient sample volume is available. Analyses of laboratory blanks are required to demonstrated freedom from contamination.

8.0 SAFETY

8.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined. Therefore, each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level.

8.2 Inhalation of hexane should be minimized by performing all operations with hexane in a hood.

8.3 Unknown samples may contain high concentrations of volatile toxic compounds. Sample containers should be opened in a hood and handled with gloves to prevent exposure.

9.0 SAMPLE PREPARATION PROCEDURES

9.1 Preparation of the analytical batch

9.1.1 Bring the analytical batch of samples, including the sample aliquots for the duplicate and MS and MSD to room temperature.

9.1.2 Place approximately 1000 mL or reagent water in a clean sample bottle to serve as the laboratory blank.

9.1.3 Prepare the LCS as described for the PAR standard (See Section 6.11). The LCS should contain hexadecane/stearic acid at a total concentration of 40-mg/L (20-mg/L of each compound).

9.1.4 Either mark the sample bottle at the water meniscus or weigh the bottle for later determination of sample volume. Weighing will be more accurate. Mark or weigh the MS and MSD.

9.2 pH verification

9.2.1 Verify that the pH of the sample is less than 2 using the following procedure

9.2.1.1 Dip a glass stirring rod into the well mixed sample

9.2.1.2 Withdraw the stirring rod and allow a drop of the sample to fall on or touch the pH paper.

9.2.1.3 Rinse the stirring rod with a small portion of hexane that will be used for extraction (to ensure that no extractable material is lost on the stirring rod). Collect the rinsate into the separatory funnel.

9.2.2 If the sample is at neutral pH, adjust the pH to <2 with HCl or H_2SO_4.

9.2.3 Add the appropriate amount of HCl or H_2SO_4 solution to the blank and LCS.

9.3 Extraction

9.3.1 <u>Preparation of the Boiling Flask</u>: Tare a clean boiling flask containing 3-5 boiling chips as follows:

9.3.1.1 Place the flask containing the chips in an oven at 105-115°C for a minimum of 2 hours to dry the flask and chips.

9.3.1.2 Remove from the oven and immediately transfer to cool to room temperature in a desiccator.

9.3.1.3 When cool, remove from the desiccator with tongs and weigh immediately on a calibrated balance.

9.3.2 Preparation of the Sample: Mark the level of sample on the outside of the sample bottle with indelible ink and transfer the entire contents to a 2-L separatory funnel. Pour the sample completely into the separatory funnel. Add 30-mL hexane to the sample bottle and seal the bottle with the original bottle cap. Shake the bottle to rinse all interior surfaces of the bottle, including the lid of the bottle cap. Pour the solvent into the separatory funnel. Do not discard the sample bottle.

9.3.3 Extract the sample by shaking the separatory funnel vigorously for 2 minutes with periodic venting into a hood to release excess pressure.

9.3.4 Allow the organic phase to separate from the aqueous phase for a minimum of 10 minutes. If an emulsion forms between the phases and the emulsion is greater than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, use of solvent phase separation paper, centrifugation, use of an ultrasonic bath with ice, addition of NaCl, or other physical methods.

9.3.5 Drain the aqueous layer (lower layer) into the original sample container. Drain a small amount of the organic layer into the sample container to minimize the amount of water remaining in the separatory funnel.

9.3.6 Place approximately 10-g anhydrous Na_2SO_4 in a filter funnel and rinse with a small portion of hexane. Discard the rinsate.

9.3.7 Drain the hexane layer (upper layer) from the separatory funnel through the Na_2SO_4 into the pre-weighed boiling flask containing the boiling chips.

9.3.8 Repeat the extraction twice more with fresh 30-mL portions of hexane, combining the extracts in the boiling flask.

9.3.9 A milky extract indicates the presence of water. If the extract is milky, allow the solution to stand for up to one hour to allow the water to settle. Decant the solvent layer through sodium sulfate to remove any excess water.

9.3.10 Measurement of the Sample Volume: Refill the sample bottle (from Section 9.3.2) to the mark with water, and then measure the volume of sample that was in the bottle, using a Class A 1-L graduated cylinder. Record the exact volume in the sample extraction logbook to a minimum of three significant numbers (e.g. 965-mL, 970-mL or 1005-mL). Use this sample volume in calculations.

10.0 EXTRACT EVAPORATION PROCEDURES

10.1 Place the weighted flask into a water bath

10.2 When the flask appears dry, remove the flask using tongs and wipe the outside surface dry to remove moisture and fingerprints.

10.3 Inspect the residue in the boiling flask for crystals. Crystal formation is an indication that sodium sulfate may have dissolved and passed into the flask. This may happen if the drying capacity of the sodium sulfate is exceeded or if the sample is not adjusted to a low pH. If crystals are observed, re-dissolve the extract in hexane, filter into another tared flask, and repeat the evaporation procedure.

10.4 Cool the boiling flask to room temperature in a desiccator and maintain in the desiccator for 30 minutes minimum. Remove with tongs and weigh immediately to determine the weight of the material in the flask.

10.4.1 If the extract was from the HEM procedure, determine the HEM (W_h) by subtracting the tare weight (Section 10.3.1) from the total weight of the flask.

10.4.2 If the extract was from the SGT-HEM procedure, (Section 10.5.5), determine the weight of SGT-HEM (Ws) by subtracting the tare weight from the total weight of the flask.

10.4.3 Determine the original sample volume in liters by filling the sample bottle to the mark with water and measuring the volume of the water in a 1 to 2L graduated cylinder. If the sample weight was used, weigh the empty bottle and cap and determine by difference, assuming a sample density of 1.00.

10.5 SGT-HEM determination

10.5.1 Silica gel capacity - To ensure that the capacity of the silica gel will not be exceeded, the amount of HEM must be less than 100 mg or if above 100 mg, must be known.

10.5.1.1 If it is known that the amount of HEM is less than 100 mg, the analysts may proceed with the determination of SET-HEM per section 10-5.3-10.5.5 without determination of HEM

10.5.1.2 If, However, the amount of HEM is not known, HEM must first be determined using the procedure in section 10.3-10.4

10.5.2 Extractable materials in silica gel - because the capacity of silica is not known for all substances, it is presumed that 3 g will absorb 100 mg of all absorbable materials. The amount of silica gel that can be used for absorption in the SGT-HEM procedure below has been limited to 30 g because of concerns about possible extractable impurities in the silica gel. Therefore, if the extract contains more than 1000 mg of HEM, split the extract per the following procedure:

10.5.2.1 Add 85-90 mL of hexane to the flask to re-dissolve the HEM. If necessary, heat the solution on a explosion proof hot plate or in a water bath to completely re-dissolve the HEM.

10.5.2.2 Transfer the extract to 1 100-mL volumetric flask. Rinse the flask sequentially with 2-3 small portions of hexane and add to the volumetric flask. Dilute to the mark with hexane.

10.5.2.3 Calculate the extract volume that contains 1000 mg of extractable material according to the following equation:

$$V_a = \frac{1000\ V_t}{W_h}$$

where:
V_a = Volume of aliquot to be withdrawn (mL)
V_t = Total volume of solvent used in section 10.5.2.2 (mL)
W_h = Weight of extractable material HEM measurement (mg)

10.5.2.4 Using a calibrated pipette, remove the volume to be withdrawn (Va) and return to the boiling flask. Dilute to approximately 100 mL with hexane.

10.5.3 Absorption with silica gel

10.5.3.1 Add 3.0 ± 0.3g of anhydrous silica gel to the boiling flask for every 100mg of HEM, or fraction thereof, to a maximum of 30 g of silica gel. For example, if the weight of HEM is 735 mg, add 3 * 8 = 24g of silica gel.

10.5.3.2 Add a fluoropolymer-coated stirring bar to the flask and stir the solution on a magnetic stirrer for a minimum of five minutes.

10.5.4 Filter the solution through hexane moistened filter paper into a pre-dried, tared boiling flask containing several boiling chips. Rinse the silica gel and filter paper with several small amounts of hexane to complete the transfer.

10.5.5 Evaporate the solution and determine the weight of SGT-HEM per section 10.4

11.0 CALCULATIONS

11.1 Hexane extractable material - Calculate the concentration of HEM ("Oil and Grease") in the sample per the following equation:

$$mg/L = \frac{W_h\ (mg)}{V_s\ (L)}$$

where:
W_h = Weight of extractable material Section 10.4.4.1 (mg)
V_s = Sample volume from Section 10.4.5 (L)

11.2 Silica gel treated hexane extractable material - Calculate the concentration of SGT-HEM ("petroleum hydrocarbons") in the sample per the equation above, substituting Ws (from section 10.4.4.2) for W_h. If the extract was split to decrease the total amount of material to 1000 mg, determine the corrected total weight of SGT-HEM in the un-split extract (W_c) using the following equation.

$$W_c\ (mg) = \frac{V_t}{V_a} * W_d\ (mg)$$

where:

Wd - Weight in the portion of the extract split for absorption
V_a = Volume of aliquot to be withdrawn (mL)
V_t = Total volume of solvent used in section 10.5.2.2 (mL)

Use the corrected total weight of SGT-HEM in the un-split extract (W_c) to determine the total SGT-HEM in the sample by substituting W_c for W_h in equation 11.1.

12.0 QUALITY CONTROL AND REPORTING

12.1 One method blank (MB) should be analyzed with each quality control (QC) batch of 20 samples or less. The MB should be free of oil and grease. Report oil&grease in the MB at levels greater than the MDL or the reporting limit (RL) as appropriate using J qualifiers if the levels are less than the method quantitation limit (MQL) in Form-1 equivalents. Report QC association of samples in EPA Form-4 equivalents. The action level for an acceptable method blank is one half of the MQL (5-mg/L). Samples associated with the method blank containing oil and grease at levels greater than one half the MQL will require re-extraction and reanalysis unless other criteria acceptable to the client are used. Since method blanks may be contaminated when they are associated with samples containing high levels of target compounds, only samples that contain less than one tenth of the MB amount may need to be re-extracted and reanalyzed. Report results to three significant figures. Do not report results below the MDL. Report results between MDL and 5.0 mg/L as estimated (with a J qualifier) if results below 5 mg/L are required for regulatory compliance.

12.2 One laboratory control sample (LCS) should be analyzed with each quality control (QC) batch of 20 samples or less. When LCS is analyzed in duplicate, the LCS or LCSD (interference-free matrices) recoveries should be within QC limits (78-114% for HEM and 64-132% for SGT-HEM). Report LCS/LCSD results in EPA Form-1 equivalents and recoveries/RPD in EPA Form-3 equivalents. See AML SOP on control charting for additional details. Contact supervisor for appropriate corrective action if LCS recoveries are outside the QC limits. Data qualification, re-extraction, and/or reanalysis of associated samples may be necessary depending on project requirements, turnaround time and other constraints.

12.3 One matrix spike (MS) should be analyzed with each quality control (QC) batch of 20 samples or less. When MS is analyzed in duplicate, the MS or MSD recoveries should be within QC limits (as shown for LCS). Flag recoveries that are outside the QC limits with an asterisk (*). Report MS/MSD results in EPA Form-1 equivalents

and recoveries/RPD in Form-3 equivalents. Contact supervisor for appropriate corrective action if DRO recoveries are outside the QC limits. Data qualification, re-extraction, and/or reanalysis of associated samples may be necessary depending on project requirements, turnaround time and other constraints.

12.4 Duplicate analyses should be performed with each QC batch of samples analyzed. Duplicate analyses may include one or more of the following: LCS duplicates, sample duplicates, or MS duplicates.

13.0References

13.1Method 1664: N-Hexane Extractable Material (HEM) and Silica gel Treated N-Hexane Extractable Material (SGT-HEM) by Extraction and Gravimetry (oil and Grease and Total Petroleum Hydrocarbons), April 1995, Document No. EPA-821-B-94-004b, EPA Water resource Center, Washington, DC.

13.2Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Petroleum Hydrocarbons, Total Recoverable, Method 418.1 (Spectrophotometric, Infrared), Issued 1978.

13.3Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Oil & Grease, Total Recoverable, Method 413.1 (Gravimetric, Separtory Funnel Extraction), Issued 1974, Editorial Revision 1978.

13.4Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Oil and Grease, Method 5520-A, 5520-E, and 5520-F.

13.5Test Methods for Evaluating Solid Waste, Volume 1C: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9070, Total Recoverable Oil and Grease (Gravimetric, Separtory Funnel Extraction), September 1986.

13.6Test Methods for Evaluating Solid Waste, Volume 1C: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9070A, n-Hexane Extractable Material (HEM) for Aqueous Samples, Update III-A and III-B.

13.7Test Methods for Evaluating Solid Waste, Volume 1C: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9071A, Oil and Grease Extraction Method for Sludge and Sediment Samples, September 1994.

13.8Test Methods for Evaluating Solid Waste, Volume 1C: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9071B, n-Hexane Extractable Material (HEM) for Sludge and Sediment Samples, Update III-A.

13.9AML Standard Operating Procedures, Q-Water-RC.

13.10 AML Standard Operating Procedures, Q-WasteMgmt-RD.

13.11 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

13.12 AML Standard Operating Procedures, Q-ControlCharts-RC.

13.13

STANDARD OPERATING PROCEDURES

No. I-OrganicNitrogen-RC

Organic Nitrogen
(EPA 350.2, 350.3, 351.2, and 351.4 and SM 4500-N_{org})

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Organic Nitrogen
(EPA 350.2, 350.3, 351.2, and 351.4 and SM 4500-N_{org})

1.0 Scope and Application

1.1 This standard operating procedure (SOP) is based on Standard Method 4500_{org}, a computational procedure for the determination of organic nitrogen.

1.2 This SOP is suitable for all concentration ranges of total Kjeldahl nitrogen (TKN) and free ammonia that are determinable by other EPA methods referenced in this SOP.

1.3 This SOP is applicable to drinking, surface, saline, and ground water.

2.0 Summary of Method

2.1 TKN and free ammonia in the water sample are determined in mg/L using applicable EPA or Standard Method referenced in this document.

2.2 The organic nitrogen in mg/L is calculated as the difference between the TKN and ammonia results.

3.0 Interferences

3.1 For specific interferences to the TKN and/or ammonia determination, see the respective SOP.
3.2 The TKN fails to account for nitrogen in the form of azide, azine, azo, hydrazone, nitrate, nitrite, nitro, nitroso, oxime, and semi-carbazone.

4.0 Sample Handling and Preservation

4.1 See SOPs for TKN and ammonia.

5.0 Calculations

5.1 Calculate organic nitrogen in mg/L from independently determined amounts (mg/L) of TKN and ammonia:

Organic Nitrogen = (TKN Result) - (Ammonia Result)

6.0 References

6.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Nitrogen, Kjeldahl, Total, Method 351.2, Colorimetric, Semiautomated Block Digestor, AAII, Issued, 1978.
6.2 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Nitrogen, Kjeldahl, Total, Method 351.4, Potentiometric, Ion selective Electrode, Issued, 1978.
6.3 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Nitrogen, Ammonia, Method 350.2 (Colorimetric, Titrimetric, Potentiometric), Distillation Procedure, Issued, 1971, and Editorial Revision 1974.
6.4 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Nitrogen, Ammonia, Method 350.3 (Potentiometric, Ion Selective Electrode), Issued, 1974.
6.5 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Method 4500-N_{org}-A and 4500-N_{org}-C.
6.6 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Method 4500-NH_3-A, 4500-NH_3-B, 4500-NH_3-E and 4500-NH_3-F.
6.7 AML Standard Operating Procedures, I-Ammonia-RA.
6.8 AML Standard Operating Procedures, I-TKN-RB.
6.9 AML Standard Operating Procedures, Q-Water-RC.
6.10 AML Standard Operating Procedures, Q-WasteMgmt-RD.
6.11 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
6.12 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-Phenolics-420_1-RC

Phenolics by Spectrophotometry (EPA 420.1, SM 5530, and SW-846 9065)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

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Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Phenolics by Spectrophotometry
(EPA 420.1, SM 5530, and SW-846 9065)

1.0 Scope and Application

1.1 This SOP is based on EPA Method 420.1, Standard Method 5530, and SW-846 Method 9065 for the determination of phenols by spectrophotometry.
1.2 This method is applicable to drinking, surface and saline waters, domestic and industrial wastes.
1.3 This method is capable of measuring phenolic materials at the 5 ug/L level when the colored end product is extracted and concentrated in a solvent pahse using pehnol as a standard.
1.4 The method is capable of measuring phenolic materials that contain more than 50 ug/L in the aqueous phase (without solvent extraction) using phenol as a standard.
1.5 It is not possible to use this method to differentiate between kinds of phenols.

2.0 Summary of Method

2.1 Phenolic materials react with 4-aminoantipyrine in the presence of potassium ferricyanide at a pH of 10 to form a stable reddish-brown colored antipyrine dye. The amount of color produced is a function of the concentration of phenolic material.

3.0 Comments

3.1 For most samples a preliminary distillation is required to remove interfering materials.
3.2 Color response of phenolic materials with 4-amino antipyrine is not the same for all compounds. Because phenolic type wastes usually contain a variety of phenols, it is not possible to duplicate a mixture of phenols to be used as a standard. For this reason phenol has been selected as a standard and any color produced by the reaction of other phenolic compounds is reported as phenol. This volue will represent the minimum concentration of phenolic compounds present in the sample.

4.0 Sample Handling and Preservation

4.1 Biological degradation is inhibited by the addition of 1 g/L of copper sulfate to the sample and acidification to a pH of less than 4 with phosphoric acid. The sample should be kept at 4 ^{o}C and analyzed within 24 hours after collection.

5.0 Interference

5.1 Interferences from sulfur compounds are eliminated by acidifying the sample to a pH of less than 4 with H_3PO_4 and aerating briefly by stirring and adding $CuSO_4$.
5.2 Oxidizing agents such as chlorine, detected by the liberation of iodine upon acidifications if the presence of potassium iodide, are removed immediately after

sampling by the addition of an excess of ferrous ammonium sulfate (7.10). If chlorine is not removed, the phenolic compounds may be partially oxidized and the results may be low.

6.0 Apparatus

6.1 Distillation apparatus, all glass consisting of a 1-Liter pyrex distilling apparatus with Graham condenser.
6.2 pH meter
6.3 Spectrophotometer, for use at 460 or 510 nm.
6.4 Funnels
6.5 Filter paper
6.6 Membrane filters
6.7 Nessler tubes

7.0 Reagents

7.1 Phosphoric acid solution, 1+9: Dilute 10 mL of 85% H_3PO_4 to 100 mL with distilled water.
7.2 Copper sulfate solution: Dissolve 100 g $CuSO_4 \bullet 5H_2O$ in distilled water and dilute to 1 liter.
7.3 Buffer Solution: Dissolve 16.9 g NH_4Cl in 143 mL conc. NH4OH and dilute to 250 mL with distilled water. Two mL should adjust 100 mL of distillate to pH 10.
7.4 Aminoantipyrine solution: Dissolve 2 g of 4AAP in distilled water and dilute to 100 mL.
7.5 Potassium ferricyanide solution: Dissolve 8 g of $K_3Fe(CN)_6$ in distilled water and dilute to 100 mL.
7.6 Stock phenol solution: Dissolve 1.0g phenol in freshly boiled and cooled distilled water and dilute to 1 Liter. Concentration = 1000 mg/mL.

8.0 Procedure

8.1 Distillation
8.1.1 Measure 50 mL sample into a beaker. Lower the pH to approximately 4 with 1+9 H_2PO_4 (7.1), add 0.5 mL $CuSO_4$ solution (7.2) and transfer to the distillation apparatus. Omit adding H_2PO_4 if sample was preserved as described in 4.1.
8.1.2 Distill 45 mL of sample, stop the distillation, and when boiling ceases, add 5 mL of warm distilled water to the flask and resume distillation until 50 mL have been collected.
8.1.3 If the distillate is turbid, filter through a prewashed membrane filter.

8.2 Direct photometric method

8.2.1 Using working solution A (7.7), prepare the following standards in 10 mL volumetric flasks.

Concentration mg Phenol / L	mL of Standard Solution / 10 mL
0.00	0.00
0.10	0.10
0.20	0.20
0.50	0.50
1.00	1.00

8.2.2 To 10 mL of distillate or an aliquot diluted to 10 mL and/or standards, add 0.2 mL of buffer solution (7.3) and mix. The pH of the sample and standards should be 10 ± 0.2.

8.2.3 Add 0.2 mL of aminoantipyrine solution (7.4) and mix.

8.2.4 Add 0.2 mL of potassium ferricyanide solution (7.5) and mix.

8.2.5 After 15 minutes read absorbance at 510 nm.

9.0 Quality Control

9.1 Method Blank should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

9.2 A Sample Duplicate should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

10.0References

10.1Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Phenolics, Total Recoverable, Spectrophotometric, Manual 4-AAP with Distillation, Method 420.1, Editorial Revision, 1978.

10.2Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Phenols, Method 5530A to 5530D.

10.3Test Methods for Evaluating Solid Waste, Volume 1C: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Phenolics, Spectrophotometric, Manual 4-AAP with Distillation, Method 9065, September 1996.

10.4 AML Standard Operating Procedures, Q-Water-RC.

10.5 AML Standard Operating Procedures, Q-WasteMgmt-RD.

10.6 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

10.7 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-Phosphorus-365_3-RC

Phosphorus by Spectrophotometry (EPA 365.3 and SM 4500-P)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

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Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Phosphorus by Spectrophotometry
(EPA 365.3 and SM 4500-P)

1.0 Scope and Application

1.1 This SOP is based on EPA Method 365.3 and Standard Method 4500-P for the determination of phosphorus in all forms.
1.2 These methods cover the determination of specified forms of phosphorus in drinking, surface and saline waters, domestic and industrial wastes.
1.3 The methods are based on reactions that are specific for the orthophosphate ion. Thus, depending on the prescribed pretreatment of the sample, the various forms may be determined.
1.4 Except for in-depth and detailed studies, the most commonly measured forms are phosphorus and dissolved phosphorus, and othrophosphate and dissolved orthophosphate. Hydrolyzable phosphorus is normally found only in sewage-type samples and insoluble forms of phosphorus are determined by calculation.
1.3 These methods are usable in the 0.01 to 1.2 mg P/L range.

2.0 SUMMARY OF METHOD

2.1 Ammonium molybdate and antimony potassium tartrate eact in an acid medium with dilute solutions of phosphorus to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the phosphorus concentrations.
2.2 Only phosphorus forms a blue color in this test. Polyphosphates (and some organic phosphorus compounds) may be converted to the orthophosphate forms by sulfuric-acid-hydrolysis. Organic phosphorus compounds may be converted to the orthophospoate form by persulfate digestion.

3.0 SAMPLE HANDLING AND PRESERVATION

3.1 If benthic deposits are present in the area being sampled, great care should be taken not to include these deposits.
3.2 Sample containers may be of plastic material, or Pyrex glass.
3.3 If the analysis cannot be performed the day of collection, the sample should be preserved by the addition of 2 mL conc. H_2SO_4 per Liter and refrigeration at 4 ^oC.

4.0 INTERFERENCES

4.1 Arsenate is determined similarily to phosphorus and should be considered when present. This interference may be eliminated by reducing the arsenic acid to arsenious acid with sodium bisulfate (6.4).

4.2 When high concentrations of iron are present low recovery of phosphorus will be obtainedbecause it will use some of the reducing agent. The bisulfate treatment will also eliminate this interference.

5.0 APPARATUS

5.1 Photometer-A spectrophometer or filter photometer suitable for measurements at 660 or 880 nm with a light path of 1 cm or longer.

5.2 Acid-washed glassware: All glassware used should be washed with hot 1:1 HCl and rinsed with distilled water. The acid-washed glassware should be filled with distilled water and treated with all the reagents to remove the last traces of phosphorus that might be absorbed on the glassware. Preferably, this glassware should be used only for the determination of phosphorus and after use it should be rinsed with distilled water and kept covered until needed again. If this is done, the treatment with 1:1 HCl and reagents in only required occasionally. Commercial detergents should never be used.

5.3 Water bath, 95°C

6.0 REAGENTS AND CHEMICALS

6.1 Ammonium molybdate-antimony potassium tartrate solution: Dissolve 0.8 g of ammonium molybdate and 0.02g antimony potassium tartrate in 80 mL of distilled water and dilute to 100 mL.

6.2 Ascorbic acid solution: Dissolve 6g of ascorbic acid in 80 mL of distilled water and tilute to 100 mL. Add 0.2 mL of acetone. This solution is stable for two weeks.

6.3 Sulfuric acid, 11N. Slowly add 31.0 mL of concentrated H2SO4 to approximately 60 mL distilled water. Cool and dilute to 100 mL.

6.4 Sodium bisulfite ($NaHSO_3$) solution: Dissolve 5.2g of $NaHSO_3$ in 100 mL of 1.0 N H_2SO_4.

6.5 Ammonium persulfate

6.6 Stock phosphorus solution: Dissolve 0.4393g of predried (105 ^{o}C for one hour) KH2PO4 in distilled water and dilute to 1000 mL. 1.0 mL = 0.1 mg P.

6.7 Standard phosphorus solution: Dilute 10 mL of stock phosphorus solution to 100 mL with distilled water. 1.0 mL - 0.01 mg P. Prepare an appropriate series of standards by dilution suitable volumes of standard or stock solutions to 100 mL with distilled water.

7.0 PROCEDURE

7.1 Total Phosphorus

7.1.1 Transfer 50 mL of sample or analiquot diluted to 50 mL into a 125 mL Erlenmeyer flask andadd 1 mL of 11N sulfuric acid (6.3).

7.1.2 Add 0.4g ammonium persulfate (6.5) , mix and boil gently for approximately 30-40 minutes or until a final volume of about 10mL is reached. Cool, dilute to approximately 40 mL and filter.

7.1.3 For samples containing arsenic or high levels of iron, add 5 mL of sodium bisulfite (6.4), mix and place in a 95 ^{o}C water bath for 30 minutes (20 minutes after the temperature of the sample reaches 95 ^{o}C). Cool and dilute to 50 mL.

7.1.4 Determine phosphorus as outlined in (7.2) orthophosphate.

7.2 Orthophosphate

7.2.1 To 50 mL of sample and/or standards, add 1 mL of 11 N sulfuric acid and 4 mL of ammonium molybdate-antimony potassium tartrate and mix. *NOTE: If sample has been digested for total phosphorus do not add acid.*

7.2.2 Add 2 mL of ascorbic acid solution and mix.

7.2.3 After 5 minutes, measure the absorbance at 650 nm with a spectrophotometer and determine the phosphorus concentration from the standard curve. The color is stable for at lease one hour.

8.0 CALCULATION

8.1 Prepare a standard curve by plotting the absorbance values of standards versus the corresponding phosphorus concentrations on linear graph paper.

8.2 Obtain concentration value of sample directly from prepared standard curve. Report results as P, mg/L.

9.0 Quality Control

9.1 A Method Blank should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

9.2 A Second Source Standard (LCS) should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

9.3 A Sample Duplicate should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

9.4 The correlation coefficient of the standard curve must be greater than or equal to 0.995.

10.0 References

10.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Phosphorus, All Forms, Method 365.3, Colorimetric, Ascorbic acid, Two Reagent, Issued 1978.

10.2 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Phosphorus, Method 4500-P.

10.3 AML Standard Operating Procedures, Q-Water-RC.

10.4 AML Standard Operating Procedures, Q-WasteMgmt-RD.

10.5 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

10.6 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-pH-Water-RC

Electrometric Measurement of pH in Water (EPA 150.1, SM 4500, and SW-846 9040B)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Electrometric Measurement of pH in Water
(EPA 150.1, SM 4500, and SW-846 9040B)

1.0 Scope and Application

1.1 This SOP is based on EPA Method 1501.1, Standard Method 4500-H^+A and B, and SW-846 Method 9040B for the determination of pH in water samples using a pH electrode.
1.2 This method is applicable to drinking, surface and saline waters, domestic and industrial wastes and acid rain (atmospheric deposition).

2.0 Summary of Method

2.1 The pH of a sample is determined electrometrically using either a glass electrode in combination with a reference potential or a combination electrode.

3.0 Sample Handling and Preservation

3.1 Samples should be analyzed as soon as possible preferably in the field at the time of sampling.
3.2 High-purity watesr and waters not at equilibrium with the atmosphere are subject to changes when exposed to the atmosphere, therefore the sample containers should be filled completely and kept sealed prior to analysis.

4.0 Interferences

4.1 The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants or high salinity.
4.2 Sodium error at pH levels greater than 10 can be reduced or eliminated by using a “low sodium error” electrode.
4.3 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by distilled water rinsing. An additional treatment with hydrochloric acid (1+9) may be necessary to remove any remaining film.
4.4 Temperature effects on the electrometric measurement of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference can be controlled with instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second source is the change of pH inherent in the sample at various temperatures. This error is sample dependent and cannot be controlled, it should therefore be noted by reporting both the pH and temperature at the time of analysis.

5.0 Apparatus

5.1 pH Meter-laboratory or field model. A wide variety of instruments are commercially available with various specifications and optional equipment.
5.2 Glass electrode.
5.3 Reference electrode - a calomel, silver-silver chloride or other reference electrode of constant potential may be used. NOTE1: Combination electrodes incorporating both measuring and reference functions are convenient to use and are available with solid, gel type filled materials that require minimal maintenance.
5.4 Magnetic stirrer and Teflon-coated stirring bar.
5.5 Thermometer or temperature sensor for automatic compensation.

6.0 Reagents

6.1 Primary standard buffer salts are available from the National Bureau of Standards and should be used in situations where extreme accuracy is necessary.
6.2 Secondary standard buffers may be prepared from NBS salts or purchased as a solution from commercial vendors. Use of these commercially available solutions, that have been validated by comparison to the NBS standardds, are recommended for routine use.

7.0 Calibration

7.1 Because of the wide variety of pH meters and accessories, detailed operating procedures cannot be incorporated into this method. Each analyst must be acquainted with the operation of each system and familiar with all instrument functions. Special attention to care of the electrodes is recommended.
7.2 Each instrument/electrode system must be calibrated at a minimum of two points that bracket the expected pH of the samples and are approximately three pH units or more apart.
7.2.1 Various instrument designs may involve use of a “balance” or “standardize” dial and/or a slope adjustment as outlined in the manufacturer’s instructions. Repeat adjustments on successive portions of the two buffer solutions as outlined in procedure 8.2 until reading are within 0.05 pH units of the buffer solution value.

8.0 Procedure and Calculations

8.1 Standardize the meter and electrode system as outlined in section 7.
8.2 Place the sample or buffer solution in a clean glass beaker using a sufficient volume to cover the sensing elements of the electrodes and to give adequate clearance for the magnetic stirring bar.
8.3 If the sample temperature differs by more than 2 ^{o}C from the buffer solution the measured pH values must be corrected. Instruments are equipped with automatic or

manual compensators that electronically adjust for temperature differences. Refer to manufacturer's instructions.

8.4 After rinsing and gently wiping the electrodes, if necessary, immerse them into the sample beaker or sample stream and stir at a constant rate to provide homogeneity and suspension of solids. Rate or stirring should minimize the air transfer rate at the air water interface of the sample. Note and record sample pH and temperature. Repeat measurement on successive volumes of sample until values differ by less than 0.1 pH units. Two or three volume changes are usually sufficient.

8.5 For acid rain samples it is most important that the magnetic stirrer is not used. Instead, swirl the sample gently for a few seconds after the introduction of the electrodes(s). Allow the electrodes(s) to equilibrate. The air-water interface should not be disturbed while measurement is being made. If the sample is not in equilibrium with the atmosphere, pH values will change as the dissolved gases are either absorbed or desorbed. Record sample pH and temperature.

8.6 pH meters read directly in pH units. Report pH to the nearest 0.1 unit and temperature to the nearest ^{o}C.

9.0 References

9.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, pH, Method 150.1, Electrometric, Editorial Revision, 1978 and 1982.

9.2 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Sulfate, Method 4500-H^{+}A and B.

9.3 Test Methods for Evaluating Solid Waste, Volume 1C: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9040B, pH, September 1986.

9.4 AML Standard Operating Procedures, Q-Water-RC.

9.5 AML Standard Operating Procedures, Q-WasteMgmt-RD.

9.6 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

9.7 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-SettleableSolids-160_5-RC

Settleable Solids
(EPA 160.5 and SM 2540F)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Settleable Solids (Method 160.5, Volumetric, Imhoff Cone)

1.0 Scope and Application

1.1 This SOP is based on EPA Method 160.5 and Standard Method 2540F for the determination of settleable solids.
1.2 The test is applicable to surface and saline waters, domestic and industrial wastes.
1.3 The MDL is estimated at 0.1-mL/L/Hour.

2.0 Summary of Method

2.1 Settleable solids is measured volumetrically using an Imhoff cone.

3.0 Comments

3.1 A separation of settleable and floating materials may occur for some samples. In such cases the floating material is not measured.
3.2 The determination of suspended solids (See AML SOP based on EPA 160.2) is preferred if floating materials should be included in the analysis.

4.0 Sample Handling and Preservation

4.1 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4 ^{o}C, to minimize microbiological decomposition of solids, is recommended.

5.0 Interferences

5.1 See comments above (Section 3.1 and 3.2).

6.0 Apparatus

6.1 Imhoff cone, 1-L
6.2 Stop watch

7.0 Procedure

7.1 Fill an Imhoff cone to the 1-L mark with a well-mixed sample. Allow the solids to settle for 45 minutes. Gently agitate sample near the sides of the cone with a rod and allow the solids to settle for 15-minutes longer.
7.2 Record the volume of solids in the cone as milliliters per liter of the sample.
7.3 If the settled matter contains pockets of liquid between large settled particles, estimate volume of these and subtract from the volume of settled solids.

8.0 Calculation

8.1 This procedure does not require any calculations to be performed. Report settleable solids as mL/L.

9.0 References

9.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Seetleable Matter, Method 160.5, Volumetric, Imhoff Cone, Issued 1974.
9.2 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Settleable Solids, Method 2540F.
9.3 AML Standard Operating Procedures, Q-Water-RC.
9.4 AML Standard Operating Procedures, Q-WasteMgmt-RD.
9.5 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
9.6 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-Sulfate-375_4-RC

Sulfate by Turbidimetry
(EPA 375.4, SM 4500, and SW-846 9038)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

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Laboratory Director, Analytical Management Laboratories Date

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Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

SULFATE BY TURBIDIMETRY

1.0 Scope and Application

1.1 This SOP is based on EPA Method 375.4 and Standard Method 4500-SO_4^{2-}E, and SW-846 Method 9038 for the determination of sulfate by turbidimetry.
1.2 This method is applicable to drinking, surface and saline waters, domestic and industrial wastes.
1.3 The method is suitable for all concentration ranges of sulfate; however, in order to obtain reliable readings, use a sample aliquot containing not more then 40mg SO_4/L.
1.4 The minimum detectable limit is approximately 1 mg/L sulfate.

2.0 Summary of Method

2.1 Sulfate ion is converted to a barium sulfate suspension under controlled conditions. The resulting turbidity is determined by a spectrophotometer and compared to a curve prepared from standard sulfate solutions.
2.2 Suspended matter and color interfere. Correct by running blanks from which the barium chloride has been omitted.
2.3 Silica in concentrations over 500 mg/L will interfere.

3.0 Comments

3.1 Proprietary reagents, such as Hach Sulfaver or equivalent, are acceptable.
3.2 Preserve by refrigeration at 4 ^{o}C.

4.0 Apparatus

4.1 Spectrophotometer for use at 450 nm with light path of 4 to 5 cm.

5.0 Reagents

5.1 *Standard:* Purchase a prepared, certified 1000 mg/L standard. -------- OR ---------
5.1.1 Standard Sulfate Solution from Na_2SO_4 (100 mg/L)
Dissolve 147.9 mg anhydrous Na2SO4 in distilled water in a 1-Liter volumetric flask and dilute to the mark with distilled water.
5.2 Barium Chloride, $BaCL_2$. Purchased from Hach. SulfaVer 4 Sulfate Reagent: Catalog Number 12065-99.
5.3 Using standard sulfate solution (5.1) prepare the following standards in 25 mL volumetric flasks. See Table in the following page:

Concentration mg Sulfate	mL of Standard Solution / 25 mL
0.00	0.00
5.0	0.125
10.0	0.250
15.0	0.375
20.0	0.500
25.0	0.625

6.0 Procedure

6.1 Use 25 mL of sample or an aliquot of sample diluted to 25 mL with distilled water, so that the concentration of sulfate does not exceed 40 mg aliquot.

6.2 Add the contents of one SulfaVer 4 Sulfate Reagent Powder pillow to the sample. Swirl to dissolve.

NOTE: A white turbidity will develop is sulfate is present.

NOTE: Accuracy is not affected by undissolved powder.

6.3 All the sample to remain undisturbed for five minutes.

6.4 Measure the absorbance at 450 nm.

7.0 Calculation

7.1 Obtain a standard curve by plotting the absorbance of standards run by the above procedure against sulfate. Compute concentration of samples by comparing sample absorbance with standard curve.

8.0 Quality Control

8.1 A Method Blank should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

8.2 A Second Source Standard (LCS) should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

8.3 A Sample Duplicate should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

8.4 The correlation coefficient of the standard curve must be greater than or equal to 0.995.

9.0 References

9.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Sulfate, Method 375.4, Turbidimetric, Technical Revision, 1978.
9.2 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Sulfate, Method 4500-SO_4^{2-}E.
9.3 Test Methods for Evaluating Solid Waste, Volume 1C: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9038, Sulfate (Turbidimetric), September 1986.
9.4 AML Standard Operating Procedures, Q-Water-RC.
9.5 AML Standard Operating Procedures, Q-WasteMgmt-RD.
9.6 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
9.7 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-Sulfide-Titrimetry-RC

Sulfide by Titrimetry
(EPA 376.1, SM 4500, and SW-846 9034)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

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Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

SULFIDE (TITRIMETRIC)

1.0 Scope and Application

1.1 This standard operating procedure (SOP) is based on EPA 376.1, Standard Method 4500-S^{2-}A/4500-S^{2-}F , and SW-846 Method 9034, which are equivalent procedures.
1.2 This SOP is applicable to the measurement of total and dissolved sulfides in drinking, surface and saline waters, domestic and industrial wastes.
1.3 Acid insoluble sulfides are not measured by the use of this test. (Copper sulfide is the only common sulfide in this class.)
1.4 This method is suitable for the measurement of sulfide in concentrations above 1 mg/L.

2.0 Summary of Method

2.1 Excess iodine is added to a sample, which may or may not have been treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is back titrated with sodium thiosulfate or phenylarsine oxide.

3.0 Comments

3.1 Reduced sulfur compounds, such as sulfate, thiosulfate and hydrosulfite, which decompose in acid may yield erratic results. Also, volatile iodine-consuming substances will give high results.
3.2 Samples must be taken with a minimum of aeration. Sulfide may be volatilized by aeration and any oxygen inadvertently added to the sample may convert the sulfide to an unmeasurable form.
3.3 If the sample is not preserved with zinc acetate and NaOH, the analysis must be started immediately. Similarly, the measurement of dissolved sulfides must also be commenced immediately.

4.0 Apparatus

4.1 Burette, pipettes, and other common laboratory glassware.

5.0 Reagents

5.1 Hydrochloric acid, HCl, 6 N
5.2 Standard iodine solution, 0.0250 N: Dissolve 20 to 25 g KI in a little water in a liter volumetric and add 3.2 g iodine. Allow to dissolve. Dilute to 1 Liter and standardize against 0.0250 N sodium thiosulfate or phenylarsine oxide using a starch indicator.
5.3 Phenylarsine oxide 0.0250 N: commercially available

5.4 Starch indicator: commercially available.

6.0 Procedure

6.1 Unprecipitated sample

6.1.1 Place a known amount of standard iodine solution (5.2) into a 500 mL flask. The amount should be estimated to be in excess of the amount of sulfide expected.

6.1.2 Add distilled water, if necessary, to bring the volume to approximately 20 mL.

6.1.3 Add 2 mL of 6 N HCl (5.1)

6.1.4 Pipette 200 mL of sample into the flask, keeping the tip of the pipette below the surface of the sample.

6.1.5 If the iodine color disappears, add more iodine until the color remains. Record the total number of milliliters of standard iodine used in performing steps 6.1.1 and 6.1.5.

6.1.6 Titrate with the reducing solution (0.0250 N sodium thiosulfate or 0.0250 N phenylarsine oxide solution (5.3)) using a starch indicator (5.4) until the blue color disappears. Record the number of milliliters used.

6.2 Precipitated samples

6.2.1 Add the reagents to the sample in the original bottle. Perform steps 6.1.1, 6.1.3, 6.1.5 and 6.1.6.

6.3 Dewatered Samples

6.3.1 Return the glass fiber filter paper, which contains the sample to the original bottle. Add 200 mL distilled water. Perform steps 6.1.1, 6.1.3, 6.1.5 and 6.1.6.

6.3.2 The calculations (7) should be based on the volume of original sample put through the filter.

7.0 Calculation

7.1 One mL of 09.0250 N standard iodine solution (5.2) reacts with 0.4 mg of sulfide present in the titration vessel.

7.2 Use the formula:

$$\text{mg/L sulfide} = \frac{400\,(A-B)}{\text{mL of sample}}$$

where:

A = mL of 0.0250 N standard iodine solution (5.2)
B = mL of 0.0250 N standard reducing sodium thosulfate or phenylarsine oxide solution (5.3)

8.0 Quality Control

8.1 A Method Blank should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.
8.2 A Second Source Standard (LCS) should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.
8.3 A Sample Duplicate should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

9.0 References

9.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Sulfide, Method 376.1 (Titrimetric, Iodine), Editorial Revision, 1978.
9.2 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Sulfide (Iodometric), Method 4500-S^{2-}A and 4500-S^{2-}F.
9.3 Test Methods for Evaluating Solid Waste, Volume 1C: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9034, titrimetric Procedure for Acid-soluble and Insoluble sulfides, December 1996.
9.4 AML Standard Operating Procedures, Q-Water-RC.
9.5 AML Standard Operating Procedures, Q-WasteMgmt-RD.
9.6 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
9.7 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-TDS-160_1-RC

Total Dissolved Solids
(EPA 160.1)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

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Laboratory Director, Analytical Management Laboratories Date

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Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Total Dissolved Solids (EPA 160.1)

1.0 Scope and Application

1.1 This SOP on the determination of total dissolved solids or filterable residue is based on EPA Method 160.1. It is applicable to drinking, surface and saline waters, domestic and industrial wastes.
1.2 The practical range of the determination is 10 mg/L to 20,000 mg/L.

2.0 Summary of Method

2.1 A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180 ^{o}C.
2.2 If Total Suspended Solids (TSS or Residue, Non-Filterable) is being determined, the filtrate from that method may be used for Total Dissolved Solids (TDS or Residue, Filterable).

3.0 Definitions

3.1 Filterable residue is defined as those solids capable of passing through a glass fiber filter and dried to constant weight at 180 ^{o}C.

4.0 Sample Handling and Preservation

4.1 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4 ^{o}C, to minimize microbiological decomposition of solids, is recommended.

5.0 Interferences

5.1 Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.
5.2 Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180°C to insure that all the bicarbonate is converted to carbonate.
5.3 Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg.

6.0 Apparatus

6.1 Glass fiber filter discs, 4.7cm or 2.1 cm, without organic binder, Reeve Angel type 934-AH, Gelman type A/E or equivalent.

6.2 Filter holder, membrane filter funnel or Gooch crucible adapter.
6.3 Suction flask, 500 mL.
6.4 Gooch crucibles, 25mL (if 2.1 cm filter is used).
6.5 125 mL Erlenmeyer flasks
6.6 Drying over, 180°C ± 2°C
6.7 Desiccator
6.8 Analytical balance, capable of weighing to 0.1 mg

7.0 Procedure

7.1 Preparation of glass fiber filter disc: Place the disc on the membrane filter apparatus or insert into bottom of suitable Gooch crucible. While vacuum is applied, wash the disc with three successive 209-mL volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Discard washings.
7.2 Preparation of Erlenmeyer Flasks: heat the clean flask to 180 ^{o}C ± 2 ^{o}C for one hour. Cool in desiccator and store until needed. Weigh immediately before use.
7.3 Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 100 mL to the funnel by means of a 100-mL graduated cylinder. If total filterable residue is low, a larger volume may be filtered.
7.4 Filter the sample through the glass fiber filter, rinse the three 10 mL portions of distilled water and continue to apply vacuum for about 3 minutes after filtration is complete to remove as much water as possible.
7.5 Transfer 100 mL of the filtrate to a weighed Erlenmeyer flask and evaporate to dryness. Dry the evaporated sample for at least one hour at 180 ^{o}C ± 2 ^{o}C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained or until weight loss is less than 0.5 mg.

8.0 Calculation

8.1 Calculate TDS as follows:

$$\text{TDS, mg/L} = \frac{(A - B) * 1000}{C}$$

where:
A = weight of dried residue + dish in g
B = weight of dish in g
C = volume of sample used in Liters

9.0 References

9.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Total Dissolved Solids, Method 160.1.
9.2 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998.
9.3 AML Standard Operating Procedures, Q-Water-RC.
9.4 AML Standard Operating Procedures, Q-WasteMgmt-RD.
9.5 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
9.6 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-Temperature-170_1-RC

Temperature
(EPA 170.1 and SM 2550)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
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Temperature (METHOD 170.1, Thermometric)

1.0 Scope and Application

1.1 This SOP is based on EPA Method 170.1 and Standard Method 2550 for the determination of temperature.
1.2 This method is applicable to drinking, surface and saline waters, domestic and industrial wastea.

2.0 Summary of Method

2.1 Temperature measurements may be made with any good grade of mercury-filled or dial type centigrade thermometer or a thermistor.

3.0 Procedure

3.1 See AML SOP on temperature measurement and control for a more detailed guidance.
3.2 Measurement device should be routinely checked against a precision thermometer certified by the National Bureau of Standards.
3.3 Report measurement results in degrees Celsius.

4.0 References

4.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Temperature, Method 170.1, Issued 1971.
4.2 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Temperature, Method 2550A and 2550B.
4.3 AML Standard Operating Procedures, Q-Water-RC.
4.4 AML Standard Operating Procedures, Q-WasteMgmt-RD.
4.5 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
4.6 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-TKN-351_3-RC

Total Kjeldahl Nitrogen (EPA 351.3 and SM 4500-N_{org})

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

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Laboratory Director, Analytical Management Laboratories Date

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Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Total Kjeldahl Nitrogen
(EPA 351.3 and SM 4500-N_{org})

1.0 Scope and Application

1.1 This SOP is based on EPA Method 351.3 and Standard Method 4500-N_{org} for the determination of Total Kjeldahl Nitrogen (TKN) in water.
1.2 This method is applicable to the determination of TKN in drinking, ground, surface and saline waters, domestic and industrial wastes.
1.3 The procedure converts nitrogen components of biological origin such as aminoacids, proteins and peptides to ammonia, but may not convert the nitrogenous compounds of some industrial wastes such as amines, nitro compounds, hydrazones, oximes, semicarbazones and some refractory tertiary amines.
1.4 The applicable range of this method is 0.1 to 20-mg/L TKN. The range may be extended with sample dilution.
1.5 TKN is defined as the sum of free-ammonia and organic nitrogen compounds, which are converted to ammonium sulfate $(NH_4)_2SO_4$ under the conditions of this procedure.
1.6 Organic Kjeldahl nitrogen (OKN) is defined as the difference obtained by subtracting the free-ammonia value from the TKN value.

2.0 SUMMARY OF METHOD

2.1 The sample is heated in the presence of sulfuric acid, K_2SO_4 and $HgSO_4$ for two and one half hours. The residue is cooled, diluted to 25-mL.
2.2 An aliquot is used for ammonia determination using one of the AML SOPa on ammonia.
2.3 An aliquot of the digested sample may also be used for phosphorus determination, when necessary.

3.0 INTERFERENCES

3.1 There are very little known interferences to this procedure.
3.2 See section 1.0 for limitations of this test procedure.

4.0 APPARATUS AND MATERIALS

4.1 Block Digester, Scientific with 40 positions.
4.2 Teflon boiling chips
4.3 Specialty glassware for digestion and distillation

5.0 REAGENTS

5.1 ASTM Type II Reagent Water (ASTM D1193). Water should be monitored for impurities including ammonia.

5.2 Concentrated Sulfuric acid, Reagent grade

5.2.1 Sulfuric acid (1:4): Dilute 10-mL of concentrated sulfuric acid with 40-mL reagent water. NOTE: Add acid to the water. Do NOT add water to the acid.

5.3 Sulfuric acid (4%): Dilute 40-mL of concentrated sulfuric acid to 800-mL with reagent water. See note above.

5.4 Potassium Sulfate (K_2SO_4), Reagent grade.

5.5 CopperSulfate ($CuSO_4.5H_2O$), Reagent grade.

5.6 **NOTE:** Skip this Section, which is included for completeness. Mercuric Sulfate: Dissolve 8.0-g red mercuric oxide (HgO) in 50-mL of 1:4 sulfuric acid and dilute to 100-mL with reagent water.

5.7 **NOTE:** Skip this Section, which is included for completeness. Digestion Solution: Dissolve 133-g of K_2SO_4 in 700-mL of reagent water and 200-mL of conc. H_2SO_4. Add 25-mL of mercuric sulfate solution and dilute to 1000-mL.

5.8 Digestion Reagent: Dissolve 134-g of K_2SO_4 (Section 5.4) and 7.3-g $CuSO_4.5H_2O$ (Section 5.5) in about 500-mL water. Carefully add 134-mL concentrtaed sulfuric acid (Section 5.2) with constant stirring. Dilute the solution to 1-L when it has cooled to room temperature. Mix well and store at room temperature.

5.9 Sodium hydroxide solution (20%): Dissolve 200-g of sodium hydroxide in 900-mL of ammonia-free reagent water and dilute to 1000-mL.

6.0 SAMPLE HANDLING AND PRESERVATION

6.1 Samples should be collected in accordance with an approved sampling plan.

6.2 Collect samples in 50-mL plastic containers with sulfuric acid preservative (to pH < 2) and analyze them as soon as possible.

6.3 Samples may be stored at 4°C and analyzed within 28 days of collection.

7.0 PROCEDURE

7.1 Pipette out 25-mL of the sample into a digestion vessel. Add 5-mL of the digestion solution (Section 5.8) and mix on a vortex mixer. Add 4 to 8 Teflon boiling chips.

7.2 With Block Digestor in the manual mode, set low and high temperatures at 160°C and preheat unit to 160°C. Place tubes in the digestor and switch to automatic mode. Set low temperature timer for 1 hour. Reset high temperature to 380°C and set timer for two and one half hours. Collect sample and dilute to 25-mL with ammonia-free reagent water.

7.3 If the digestate is not used for any other analyses (e.g. Phosphorus) use the entire digestate (25-mL) for ammonia determination. Adjust the pH to 9.5 with sodium hydroxide solution, add borate buffer distill off ammonia into a 2% boric acid solution. Adjust the final volume of the distillate to 25-mL with ammonia-free reagent water.

7.4 See applicable SOP on ammonia determination. Ammonia determined spectrophotometrically equals TKN.

8.0 CALCULATIONS

8.1 Calculate TKN by including appropriate dilution factors if the entire digestate is not used for ammonia determination. Dilution factor also needs adjustment if the final volume of the boric acid distillate is different from the initial sample volume.

8.2 Obtain a standard curve by plotting the absorbance of standards run by the above procedure against NH_3-N (mg/L).

8.3 Compute concentration of samples by comparing sample absorbance against the standard curve.

9.0 Quality Control

9.1 A Method Blank should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

9.2 A Second Source Standard (LCS) should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

9.3 A Sample Duplicate should accompany every batch of samples analyzed, or every 20 samples, whichever is greater.

10.0 References

10.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Nitrogen, Kjeldahl, Total, Method 351.2, Colorimetric, Semiautomated Block Digestor, AAII, Issued, 1978.

10.2 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Nitrogen, Kjeldahl, Total, Method 351.4, Potentiometric, Ion selective Electrode, Issued, 1978.

10.3 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Nitrogen, Ammonia, Method 350.2 (Colorimetric, Titrimetric, Potentiometric), Distillation Procedure, Issued, 1971, and Editorial Revision 1974.

10.4 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Nitrogen, Ammonia, Method 350.3 (Potentiometric, Ion Selective Electrode), Issued, 1974.

10.5 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Method 4500-N_{org}-A and 4500-N_{org}-C.

10.6 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Method 4500-NH_3-A, 4500-NH_3-B, 4500-NH_3-E and 4500-NH_3-F.

10.7 AML Standard Operating Procedures, Q-Water-RC.

10.8 AML Standard Operating Procedures, Q-WasteMgmt-RD.

10.9 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

10.10 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-TOC-RC

TOTAL ORGANIC CARBON

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) for the determination of total organic carbon (TOC) is based on EPA Methods SW-846 9060 and 415, and Standard method 5310. This SOP is applicable to the measurement of TOC in a variety of water samples (drinking, ground, saline and surface water), domestic and industrial wastes, and soil samples. This document also includes procedures for the determination of inorganic carbon (IC) and other types of carbon such as dissolved organic carbon (DOC) and total carbon (TC).

1.2 The final usefulness of TOC measurement is in assessing the potential oxygen-demanding load of organic material on a receiving system such as a stream or groundwater. This statement applies whether the carbon measurement is made on a sewage plant effluent, industrial waste, industrial waste, and groundwater or on water taken directly taken from the stream. In this light, carbonate, bicarbonate, carbonic acid, and dissolved carbon dioxide carbon are not a part of the oxygen demand in the stream and therefore should be discounted in the final calculation or removed prior to analysis. The manner of preliminary treatment of the sample and instrument settings defines the types of carbon, which are measured.

1.3 Dissolved organic carbon (DOC) may be measured by filtering the sample using a 0.45-μ filter prior to determination of total organic carbon (TOC).

2.0 SUMMARY OF METHOD

2.1 Carbon is measured using a commercially available carbonaceous analyzer instrument (Tekmar Dohrmann Model Apollo 9000). This instrument converts the carbon in a sample to carbon dioxide (CO_2) by high-temperature combustion in accordance with EPA Method 415.1 or SM 5310B. The CO_2 formed is then measured directly by a using a non-dispersive infrared detector (NDIR) after removing moisture using a trap and halogens using a corrosives scrubber containing Pyrex wool, tin and copper granules. The amount of CO_2 in a sample is directly proportional to the concentration of carbonaceous material in the sample.

2.2 Carbonaceous analyzers are capable of measuring all forms of carbon in a sample. However, because of various properties of carbon-containing compounds in liquid samples, the manner of preliminary sample treatment as well as the instrument settings will determine which forms of carbon are actually measured. The forms of carbon that can be measured by this SOP are:

- 2.2.1 Soluble, nonvolatile organic carbon: e.g., natural sugars
- 2.2.2 Soluble, volatile organic carbon: e.g., mercaptans, alkanes, low molecular weight alcohols, small amounts of chlorinated solvents, etc. in the absence of inorganic carbon
- 2.2.3 Insoluble, partially volatile carbon: e.g., low molecular weight oils.
- 2.2.4 Insoluble, particulate carbonaceous materials: e.g., cellulose fibers.
- 2.2.5 Soluble or insoluble carbonaceous materials adsorbed or entrapped on insoluble inorganic suspended matter: e.g., oily matter adsorbed on silt particles.

2.3 Total carbon (TC) includes all the carbon present in the sample: organic carbon (OC) and inorganic carbon (IC). Sample is delivered directly to the combustion furnace without pretreatment.

2.4 Carbonates, bicarbonates, carbonic acid, and dissolved carbon dioxide are inorganic forms of carbon (IC), which are measured by acidifying aqueous samples to a pH less than 3 and sparging the derived carbon dioxide (CO_2) through the detector. The purging procedure also removes purgeable organic carbon (POC) from the sample. The IC data for samples containing high levels of volatile organic compounds (VOCs) will be biased higher by the POC.

2.5 Organic carbon (OC) is a measure of all carbon atoms covalently bonded in organic molecules. The OC measurement requires the separation of inorganic forms of carbon from the total organic carbon value. Depending on the instrument manufacturer's instructions, this separation can be accomplished by either a simple mathematical subtraction (TC-IC Method, Measurement by difference) or by actual removal of the carbonate and bicarbonate (TOC Method, Measurement by IC removal) from the sample prior to analysis. The Apollo 9000 performs these two types of TOC analysis automatically.

2.5.1 The TOC Method for aqueous samples involves IC removal from samples using either the IC sparger or the auto sampler module (ASM) sparger.

2.5.1.1 In the ASM sparge method, acid is delivered to the sample vial contained in ASM. The sample is sparged in the vial using a needle sparger and a known aliquot transferred to the combustion chamber.

2.5.1.2 In the IC sparge method, a known aliquot of the sample is delivered to the IC sparger, contained in Apollo 9000. The acid is delivered to the IC sparger and the sample is sparged using a frit in the IC sparger and the treated sample is transferred to the combustion furnace. ***NOTE:*** *The IC sparge is the preferred mode of operation since more complete IC removal is achieved in this mode.*

2.5.2 The DOC method for aqueous samples involves TOC analysis on water samples that have been filtered using a 0.45-µ filter either in the field or in the laboratory.

2.5.3 The TOC method for soil samples involves TOC analysis using a boat sampler (Model 183 boat sampling module) on soil samples that have treated with acid to remove IC.

2.6 Suspended organic carbon or particulate organic carbon refers to the fraction of TOC retained by a 0.45-µ filter (Section 2.5.2). This is measured using a boat sampler, when required.

3.0 INTERFERENCES

3.1 Carbonate and bicarbonate carbons represent interference for the determination of organic carbon and must be removed or accounted for in the final calculation. In most water samples, IC fraction is many times greater than the TOC fraction. Therefore, complete removal of IC is required for accurate and reproducible TOC results.

3.2 The TOC procedure for aqueous samples is applicable only to homogeneous samples, which can be injected into the apparatus reproducibly by means of a microliter-type syringe or pipet. The openings of the syringe or pipet limit the maximum size of particle, which may be present in the sample.

3.3 Removal of carbonate and bicarbonate by acidification and purging with nitrogen, or other inert gas, can result in the loss of volatile organic substances. Therefore, organic carbon measurement made after eliminating IC is actually a non-purgeable organic carbon determination.

3.4 The high temperature combustion method employed in this SOP will determine organic carbon from compounds that are chemically refractory and not determined by other oxidation methods such as persulfate-UV or heated persulfate method (SM 5310C or EPA 415.2) and wet oxidation method (SM 5310D). Therefore, biases on the order of 1-mg/L for the combustion method are not uncommon.

3.5 The method described in this SOP is highly desirable for samples containing high levels of carbon such as wastewater, which may contain TOC in excess of 100-mg/L. Drinking water TOCs can range from 0.1-mg/L to >25-mg/L. This SOP will produce data with acceptable detection limit levels for drinking water and groundwater.

4.0 APPARATUS AND MATERIALS

4.1 Tekmar-Dohrmann Apollo 9000 TOC Combustion Analyzer
4.2 Tekmar-Dohrmann STS 8000 9000 TOC auto sampler module (ASM)
4.3 Tekmar-Dohrmann 183 TOC Boat Sampling Module
4.4 Waring blender for blending or homogenizing samples
4.5 VOA vials, 40-mL
4.6 Volumetric flasks, 50-mL, and 100-mL
4.7 Eppendorf pipettes, 10 to 100-uL and 100 to 1000-uL
4.8 Pipette Tips
4.9 Balance - Analytical, 0.0001 grams and top loading, 0.1 grams
4.10 Stainless steel table spoons
4.11 Porcelain dish
4.12 Mortar and pestle

5.0 REAGENTS

5.1 ASTM Type I water (ASTM D1193): Water should be monitored for impurities, and should be boiled and cooled to remove CO_2. See applicable AML SOP regarding reagent water.

5.2 TOC Stock standard - Obtain certified TOC standard from the instrument manufacturer (Tekmar-Dohrmann P/N 511-947). This standard contains TOC at a concentration of 2000-mg/L. The carbon is present in the form of potassium hydrogen phthalate. Store standards in glass containers at 4°C. This standard expires from the date of preparation or as stated by the manufacturer, whichever is earlier.

5.3 Second source TOC standard - Potassium hydrogen phthalate, stock solution, 1,000-mg/L carbon: Dissolve 0.2128 g of potassium hydrogen phthalate (primary standard

grade) in carbonate-free Type I water and dilute to 100-mL. Alternately, acquire this compound from EM Science (cat # PX 1476-1). NOTE: Sodium oxalate and acetic acid are not recommended as stock solutions. Plastic containers and Parafilm may contain high levels of phthalate esters, which will bias sample results. Plastic should be avoided to the extent possible.

5.4 Aqueous TOC Initial Calibration Standard Solutions (ICAL): Prepare a series of ICAL solutions fresh each day of analysis from the stock solution by dilution with Type I water. Use glass containers only (no plastics) and Teflon-lined caps to prepare standards. Prepare 1-mg/L, 5-mg/L, 10-mg/L, 15-mg/L, and 20-mg/L solutions. Prepare 50-mL of the 1-mg/L solution (ICAL-1) by diluting 25-mL of the 2000-mg/L TOC stock standard solution (Section 5.2) to 50-mL. Prepare 1-mg/L solution by diluting 25-uL of the stock standard (Section 5.2) to 50-mL with Type I water. Use the following amounts for the other ICAL standards: ICAL-2 (5-mg/L) - 125-uL to 50-mL, ICAL-3 (10-mg/L) 250-uL to 50-mL, ICAL-4 (15-mg/L) - 375-uL to 50-mL, and ICAL-5 (20-mg/L) - 500-uL to 50-mL. Transfer ICAL standards to clean 40-mL VOA vials for analysis.

5.5 Blanks - Use 50-mL of the Type I water used to prepare ICAL solutions as initial calibration blank (ICAL-0), initial calibration verification blank (ICB) and continuing calibration verification blanks (CCB). Analyze 40-uL of the blank water contained in the boat sampler for soil blanks.

5.6 Aqueous Initial Calibration Verification (ICV) standard for TOC analysis. This is prepared using a second-source standard (Section 5.3). Prepare 10-mg/L TOC solution by diluting 500-uL of the 1000-mg/L second source standard (Section 5.3) to 50-mL with Type I water. Prepare fresh every day.

5.7 Aqueous Continuing Calibration Verification (CCV) standard for TOC analysis. This is prepared using the stock standard (Section 5.2). Prepare 10-mg/L TOC solution by diluting 250-uL of the 2000-mg/L standard (Section 5.2) to 50-mL with Type I water. Prepare fresh every day.

5.8 Aqueous LCS and MS spiking solution - The stock standard (Section 5.2) is used for preparing spiked samples. Blanks and samples are spiked at 10-mg/L.

5.8.1 Prepare 10-mg/L TOC solution for LCS and LCSD analysis by diluting 250-uL of the 2000-mg/L stock standard (Section 5.2) to 50-mL with Type I water. Prepare fresh every day. Transfer the standard to a 40-mL VOA vial for analysis.

5.8.2 Prepare spiked (10-mg/L default) samples for MS and MSD analysis by diluting 250-uL of the stock 2000-mg/L standard (Section 5.2) to 50-mL with the sample requiring MS/MSD analysis. Dilute sample with appropriate volumes of the Type I water if the sample requires dilution to keep the MS and MSD amounts within the calibration range. Transfer the standard to a 40-mL VOA vial for analysis.

5.9 Soil TOC Initial Calibration Standard Solutions (ICAL): Prepare a series of ICAL solutions fresh each day of analysis from the stock solution by dilution with Type I water. Use screw-cap GC vials to prepare small quantities of soil ICAL standards. Prepare 20-mg/L, 50-mg/L, 200-mg/L, 500-mg/L, and 2000-mg/L solutions as described below:

5.9.1 2000-mg/L - Use the stock standard (Section 5.2) without dilution.
5.9.2 500-mg/L - Dilute 250-uL of the stock standard to 1-mL with Type 1 water.
5.9.3 200-mg/L - Dilute 100-uL of the stock standard to 1-mL with Type 1 water.
5.9.4 50-mg/L - Dilute 25-uL of the stock standard to 1-mL with Type 1 water.
5.9.5 20-mg/L - Dilute 10-uL of the stock standard to 1-mL with Type 1 water.
5.9.6 Transfer 40-uL of each standard ICAL standard to the boat sampler for analysis. The boat sampler can contain 100-mg of treated silica gel (Section 5.16) at the analyst's option. The micrograms of soil analyzed under ICAL are as follows: 0.8, 2.0, 8.0, 20, and 80. The manufacturer recommended minimum and maximum calibration amounts are 0.5 and 160-µg carbon respectively.

5.10 Soil Initial Calibration Verification (ICV) standard for TOC analysis. This is prepared using a second-source standard (Section 5.3). Prepare 1-mL of a 200-mg/L TOC solution by diluting 200-uL of the 1000-mg/L second source standard (Section 5.3) to 1.0-mL with Type I water. Prepare fresh every day. Analyze 40-uL of this standard as soil ICV. Treated (See Section 5.16) silica gel (100-mg) may be included in the boat sampler.

5.11 Soil Continuing Calibration Verification (CCV) standard for TOC analysis. This is prepared using the stock standard (Section 5.2). Prepare 1-mL of a 200-mg/L TOC solution by diluting 100-uL of the 1000-mg/L standard (Section 5.2) to 1.0-mL with Type I water. Prepare fresh every day. Analyze 40-uL of this standard as soil CCV. Treated (See Section 5.16) silica gel (100-mg) may be included in the boat sampler.

5.12 Aqueous LCS and MS spiking solution - The stock standard (Section 5.2) is used for preparing spiked samples. Blanks and samples are spiked at 10-mg/L to prepare LCS and MS samples respectively.

5.12.1 The soil LCS standard solution is identical to the 200-mg/L soil CCV standard. Prepare LCS by spiking 40-uL of this standard to 100-mg of treated silica gel (Section 5.16) contained in the boat sampler. The silica gel may be omitted if blank values for silica gel are unacceptable. The default soil LCS concentration is 80-mg/kg.

5.12.2 The soil MS standard solution is identical to the 200-mg/L soil CCV standard. Prepare MS (and MSD) by spiking 40-uL of this standard to 100-mg of the pre-treated sample selected for matrix spike analysis (See Section 9.1.2) contained in the boat sampler. The default soil MS concentration is 80-mg/kg. Dilute sample with appropriate volumes of treated silica gel (Section 5.16) if the sample requires dilution to keep the MS and MSD amounts within the calibration range.

5.13 IC Stock standard - Prepare carbonate-bicarbonate stock solution (1,000 mg/L carbon) by dissolving 0.3500-g of sodium bicarbonate and 0.4418-g of sodium carbonate in Type I water and diluting it to 100-mL in a volumetric flask. Prepare fresh every day.

5.14 Prepare initial calibration standards (ICAL) as described in Section 5.4, calibration verification standards (CCV) as described in Section 5.7 and LCS/MS samples as described in Section 5.8. Use double the amount of stock standard shown in the referenced sections since the stock IC standard (1000-mg/L C) is prepared at one half the concentration of the stock TOC solution (2000-mg/L C). Analysis for IC is seldom required.

5.15 Phosphoric acid (cat # 0260-02) from J.T. Baker.

5.15.1 Prepare 1:1 solution by diluting 50-mL of 85% H_3PO_4 to 100-mL with Type I water. Use it for pre-treating soil samples.

5.15.2 Prepare 1:4 solution by diluting 50-mL of 85% H_3PO_4 to 200-mL with Type I water. Use it as the acid reagent for Apollo 9000 instrument, which is used with IC and/or ASM sparger.

5.16 Silica gel (200 mesh). Pre-ignite by ramping to 850°C in a muffle furnace prior to use.

5.17 Zero grade air (99.98%) containing less than 1-ppm carbon dioxide. Obtain as a cylinder from a gas supplier.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Aqueous samples for TOC and/or DOC analysis- Containers commonly used for collecting aqueous samples for VOCs analysis (40-ml VOA vials) are suitable for collecting samples for TOC and/or DOC analysis. Provide three (3) vials containing the acid (H_2SO_4) preservative for each water sample to be collected. Although plastic containers are permissible, they are not desirable for samples containing very low levels of carbon. Upon receipt and log-in, the sample containers should be stored in the walk-in cooler at 4°C and protected from sunlight and atmospheric oxygen until analysis.

6.1.1 Samples requiring dissolved organic carbon (DOC) analysis require filtration through 0.45µ filter. If samples have been filtered in the field, analyze samples as received and report the results as DOC. Otherwise, filter the sample in the laboratory through a 0.45µ filter prior to analysis.

6.1.2 Quality control samples such as MS and MSD, and field blanks should be treated as regular environmental samples. Provide one set of containers for each such sample.

6.2 Aqueous samples for inorganic carbon (IC) - Provide three (3) vials with no added preservatives for each water sample to be collected. Acid preservation invalidates any inorganic carbon determination on the samples. Analysis for IC is seldom required.

6.3 Soil samples for TOC analysis - Provide one 2-Oz clear wide mouth jar for each sample to be collected. Provide an additional jar for each QC sample (sample duplicate, MS, MSD, etc.) that needs to be analyzed. Sample containers should be stored in the walk-in cooler at 4°C and protected from sunlight and atmospheric oxygen until analysis.

6.4 Water samples should be analyzed for TOC within the holding times, which is 28-days from the date of collection for acid-preserved samples.

6.5 Soil samples should be analyzed for TOC within the holding times, which is 28-days from the date of collection.

7.0 HEALTH AND SAFETY

7.1 Calibration standards and other standards used for quality control may contain acids, bases and/or toxic compounds such as phthalate esters. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.2 Samples may contain high levels of organic and/or inorganic contaminants. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

7.3 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

7.4 Proper sample disposal practices (see AML SOPs) should be followed to minimize exposure to toxic compounds.

8.0 WASTE DISPOSAL

8.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see the AML SOP on waste disposal.

8.2 For procedures on dealing with expired standards, see the AML SOP on waste disposal.

9.0 PROCEDURE

9.1 Sample Preparation

9.1.1 Aqueous samples - Most aqueous samples do not require additional sample preparation unless they (e.g. wastewater) contain large particulates. Samples requiring homogenization may include wastewater samples. Homogenize such samples in a blender to reduce particle size so that the sample syringes can handle them without getting clogged. If the samples were not preserved with acid at the time of sample collection, lower the pH of the sample to 2. Transfer the samples to 40-mL VOA vials and load them on the autosampler. The instrument uses 0.5-mL of the sample for analysis.

9.1.2 Soil and sediment samples - The inorganic carbon (IC) should be removed as described below prior to TOC analysis as described below. Weight out approximately 10-g of the solid sample into a glass or porcelain vessel. Homogenize dry samples to a powder, if possible with a pestle. Add 10-mL of the 1:1 phosphoric acid (Section 5.11) and stir the slurry with a glass rod. Place the slurry in an oven overnight at 75°C or until dry. Cool the soil sample and ground to a fine powder with a pestle. Store the sample in a properly labeled scintillation vial. Use 100-mg aliquots of the treated sample for total organic carbon (TOC) analysis. Use the untreated sample for total carbon (TC) analysis. Analyze smaller sample quantities (e.g. 10 to 100-mg) if calibration limits are exceeded.

9.2 Instrument Calibration

9.2.1 Follow instrument manufacturer's instructions (see Appendix 3 for a summary) for instrument setup, calibration and calculations. The Apollo 9000 instrument (See Section 4.1 to 4.3 and Section 12.5 to 12.6) can be

operated at several modes:

9.2.1.1 TOC mode in the following concentration ranges: 0 to 20-ppm, 1to 400-ppm, 10 to 750-ppm, 100 to 4000-ppm, and 1000 to 25,000-ppm. The most sensitive range (0 to 20-ppm) is used for routine TOC analysis. Use a wider range (e.g. 1 to 400-ppm range) for wastewater samples. The instrument will report total organic carbon (TOC) results. The TOC values should be interpreted as dissolved organic carbon (DOC) if filtered samples are used for analysis. NOTE: The calibration scheme may need to be modified if ranges other than 0 to 20-ppm are used.

9.2.1.2 TC mode is also available in the concentration ranges shown in Section 9.2.1.1. The IC removal step is omitted for this analysis. It is preferable to have unpreserved samples for this analysis.

9.2.1.3 TC-IC mode is an alternate mode for TOC analysis, which is also available in the concentration ranges shown as in Section 9.2.1.1. The TC-IC mode should not be used if the IC concentration is greater than 50% of the TC. Since most groundwater samples may contain more IC than OC, the TOC mode is the preferred method.

9.2.1.4 IC mode, which is also available in the concentration ranges shown as in Section 9.2.1.1. Do not use acid-preserved samples for this analysis.

9.2.1.5 Boat Sampler mode - This mode is normally used for solid sample analysis. The boat sampler can handle 10 to 100-mg sample and 5 to 40-µL of liquid samples or standards. The standard carbon range is from 0.5 to 160-µg.

9.2.2 Calibration for Aqueous Samples - For calibration of the instrument, a series of standards (See Section 5.4 and 5.5) should be used that encompasses the expected concentration range of the samples. Load aqueous calibration standards (Section 5.4) contained in 40-mL VOA vials in the autosampler module or the soil ICAL standards (Section 5.9) into the boat sampler in sequence. Section 4.3 of the instrument manual (See Section 12.5) should also be consulted for details on the instrument software. Use four (4) replicates for the analysis of each standard. The samples should also be analyzed using four (4) replicates. The calibration curve screen appears when the standard run ends. It displays the calibration factor or slope (m), y-intercept (b_{cal}), correlation coefficient (r^2) for the linear regression (see Appendix 1 for details). It also displays the expected and measured concentration calculated using the linear regression for each of the calibration standards. The instrument uses only the slope (m) as the calibration factor to calculate measured concentrations. The y-intercept (b_{cal}) is not used since the carbon associated with a blank may change over time. Special blank methods are used to determine to update the y-intercept (b_{new}) for sample calculations. The correlation coefficient for the plot of response against concentration should be greater than 0.995 for the initial calibration (ICAL)

to be acceptable.

9.2.3 Calibration for soil samples - Prepare soil ICAL standards as described in Section 5.9. Pipette 40-uL of each ICAL standard including the ICAL blank into the boat sampler containing 100-mg of treated (Section 5.16) silica gel. The silica gel may be omitted at the discretion of the analyst.

9.2.4 ICV - Verify the multi-point initial calibration (ICAL) curve immediately after the analysis of the ICAL solutions by analyzing a second-source initial calibration verification standard (ICV) prepared as described in Section 5.6 for aqueous samples and Section 5.10 for soil samples. If the response varies from the expected value by more than ± 15%, the ICAL is not acceptable. A third source ICV standard and/or a freshly prepared second-source ICV should be used to verify ICAL. If the ICV fails, the ICAL standards should be replaced and the ICV should be repeated until ICV is within ± 15% of the true value.

9.2.5 CCV - The initial calibration curve (ICAL) should also be verified (CCV) on each working day prior to the analysis of any sample. Repeat CCV after the analysis of every 15 samples. Continuing calibration verification standards are (CCV) are prepared as described in Section 5.7 for aqueous samples and Section 5.11 for soil samples. If the response varies from the expected values by more than ± 10%, the CCV should be repeated, using fresh calibration standards. If the results are still more than ± 20%, a new calibration curve (ICAL) should be prepared. Document CCV in instrument printouts or Form-7 equivalents (see Appendix 1 for details).

9.2.6 ICB and CCB - Analyze initial and continuing calibration blanks immediately after the analysis of ICV and CCV standards. The blank level should be less than one half of the applicable method quantitation limit (MQL). Blank samples for water and soil matrices are prepared as shown in Section 5.5.

9.3 Sample Analyses

9.3.1 Establish method detection limits (MDL) and method quantitation limits (MQL) for each analyte and for each matrix as described in applicable AML SOPs (Section 12.9 and 12.10).

9.3.2 Select instrument mode as described in Section 9.2.1 and establish an initial calibration curve (ICAL) as described in Section 9.2.2. Verify calibration as described above (Section 9.2.3 to 9.2.5).

9.3.3 Prepare soil and water samples for analysis as described in Section 9.1. Prepare soil and water MB, LCS, LCSD, MS, and MSD samples as described in various subsections under Section 5.

9.3.4 Analyze MB, LCS, and LCSD samples. Calculate the concentration from the calibration curve and compare the results to their known values. If the quality control limits are exceeded, stop the analysis, identify and correct the problem. Re-calibration (new ICAL) of the instrument may be necessary.

9.3.5 Analyze the sample designated for matrix spike, the matrix-spike sample (MS) and the MS duplicate (MSD). Repeat sample, MS and MSD if the

calibration limits are exceeded.

9.3.6 Analyze samples associated with the QC batch ensuring that CCVs and CCBs are analyzed at the proper frequencies as described in Section 9.2.4 and 9.2.5.

9.3.7 Please note that four replicate analyses of the samples are required. Report the mean result for sample data. Determine the range (high and low) and standard deviation for the quadruplicate analysis. Report these parameters, if required.

9.4 Calculations

9.4.1 Verify instrument printouts independently by entering the calibration standard concentrations and instrument responses from instrument printouts into an Excel spreadsheet (see Appendix 2 for details).

9.4.2 Calculate the following parameters: slope (m), intercept (c), and correlation coefficient (r). The slope and intercept define a relationship between the concentration and instrument response of the form:

$$y_i = m\, x_i + b \qquad (1)$$

where:
y_i = Instrument response in the standard
m = response slope
x_i = concentration of standard
b = y-intercept

Rearrangement of the above equation yields the concentration corresponding to an instrumental measurement:

$$x_j = (y_j - b)/m \qquad (2)$$

where:

x_j = calculated concentration for a sample
y_j = actual instrument response for a sample
m and b are calculated slope and intercept from calibration above.

9.4.3 Enter the sample responses into an Excel spreadsheet, and calculate the sample concentration in milligrams per liter. Report the mean result if quadruplicate measurements are made.

9.4.4 For solid samples, multiply the mg/L results obtained by the volume of sample extract (ml) and divide by the weight (g) of the solid sample analyzed.

10.0 QUALITY CONTROL

10.1 Quality control procedures are used to ensure that proper sample preparation techniques and instrument calibration procedures are performed. This involves the analysis of method blanks (MB), laboratory control samples (LCS), LCS duplicates (LCSD), sample duplicates, matrix spikes (MS), and MS duplicates (MSD).

10.2 The frequency of analysis of LCS analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples. LCS can be run in duplicate to obtain precision (RPD) information. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. See Tables 1 and 2 for QC limits.

10.3 The frequency of analysis of MS analysis is equivalent to a minimum of 2 per 20 samples or 2 per batch if less than 20 samples. MS is run in duplicate to obtain precision (RPD) information. See Tables 1 and 2 for QC limits.

10.4 The frequency of analysis of MB analysis is equivalent to a minimum of 1 per 20 samples or 1 per batch if less than 20 samples.

10.5 Analyze a second source initial calibration verification standard (ICV) immediately after multi-point initial calibration (ICAL). The calculated values (or instrument response) for the ICV should be within ±15 percent of the true values (or response from initial calibration). When the ICV is outside the acceptance window, the laboratory should take corrective action with ICAL and/or ICV standards.

10.6 Include a calibration verification standard (CCV) after each group of 15 samples in the analysis sequence as a calibration check. The calculated values (or instrument response) for the calibration verification should be within ±20 percent of the true values (or response from initial calibration). When this continuing calibration is out of this acceptance window, the laboratory should stop analyses and take corrective action.

10.7 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair, spiked with the analyte at the mid point of initial calibration curve. If samples are expected to contain target analytes, then the laboratory may use one matrix spike and a duplicate analysis of an unspiked field sample. Since this knowledge may not always be available, AML will always analyze the MS in duplicate (MS and MSD). Sample duplicate analyses need not be performed unless client requests duplicate analysis for a specific project. Duplicate analyses should be performed if the sample selected for MS contains large amounts of total carbon.

10.8 For TOC analysis on aqueous groundwater samples, manually pre-purge at least one acidified sample for ten minutes with pure nitrogen and analyze it as a QC

duplicate sample to check on the instrument's IC removal efficiency. If the sample result for the pre-purged sample is less than 80% of the value obtained when the instrument's the IC sparger alone is used, stop the analysis, correct the problem and re-analyze all associated samples.

10.9 Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

11.0DATA VERIFICATION AND VALIDATION - This section will provide guidance when evaluating and validating data generated by this SOP. It provides procedures to assess if data produced is valid and legally defensible. Establish method detection limits (MDL) and method quantitation limits (MQL) for the analysis (see Appendix 9 for details). Document instrument printouts in project files and report sample data in Form-1 equivalents (see Appendix 7 for details). Document review on a checklist (see Appendix 6 for details) and on the case narrative as appropriate. Document association of samples with quality control analyses in Form-4 equivalents (see Appendix 4 for details). Clients such as US Army Corps of Engineers (USACE) and air force (AFCEE) may have special requirements, which are documented in method quality objectives (MQOs). Contact your supervisor (see also Appendix 10 for details) for more information.

11.1HOLDING TIMES – The validity of analytical data is based on holding times of the samples from the time of collection to the time of analysis or sample prep.

11.1.1 WATER - The holding time for this analysis is 28-days. Holding times are calculated in days from the date of sampling. Holding times are established by comparing the information in the chain of custody/sample log-in information with the date of the injection.

11.1.2 SOIL / SOLIDS– The holding time for this analysis is 28-days for soil and waste samples.

11.1.3 ACTION – If holding times have been exceeded, contact the client. All positive results may require qualification as estimated (J) by the user (client). Include a statement in the case narrative indicating the outlier. If the holding times are greatly exceeded, then the reviewer (client) may use professional judgement and may determine sample results to be unusable, rejected (R) or invalid (I).

11.2INITIAL CALIBRATION (ICAL) – Instrument calibration criteria are established to ensure that the instrument is capable of producing acceptable quantitative data. The ICAL demonstrates that the instrument is capable of producing data within a range that is linear.

11.2.1 CHECK – Check to see that the correlation coefficient is greater than 0.995.

11.2.2 ACTION – Reanalyze the affected standard(s) if the one or more standards exhibit values that deviate significantly from the expected or true values. Check the instrument for proper operation. Prepare a fresh

set of ICAL solutions and repeat the initial calibration procedure. If the situation cannot be corrected within the holding time of the samples, perform emergency sample analysis using less than 5 calibration points for the ICAL. Positive results not meeting the correlation coefficient criteria may need to be qualified as estimated (J) by the user (client). Report detects and non-detects without qualifiers and mention these outliners in the case narrative.

11.3 INITIAL CALIBRATION VERIFICAION (ICV) – ICV using a second-source is performed to verify the integrity of the calibration standards used for ICAL.

11.3.1 CHECK – Check to see that the observed value is within ±15% of the true value.

11.3.2 ACTION - Correct the problem and rerun the calibration verification and all associated samples. Positive results for compounds not meeting the criteria may be qualified as estimated (J) by the user (client). Report non-detects without qualifiers and mention these outliners in the case narrative.

11.4 CONTINUING CALIBRATION VERIFICATION (CCV) – The continuing calibration is used to check that the instrument is still operating under the linear range of the initial calibration and has remained stable. The calibration is verified after every 15 sample injections. Document ICV and CCV results and %Ds in Form-7 equivalents.

11.4.1 CHECK – Check to see that the observed value is within ±20% of the true value.

11.4.2 ACTION – Correct the problem and rerun the calibration verification and all associated samples. Re-analyze affected samples if holding times are not exceeded. Positive results for compounds not meeting the criteria may be qualified as estimated (J) by the user (client). Report non-detects without qualifiers and mention these outliners in the case narrative.

11.5 METHOD BLANKS – The assessment of method blank analysis is to determine the degree of potential contamination introduced during various sample-processing procedures, from the field to the laboratory manipulations. Sample data qualification to alert clients about blank contamination is performed by the laboratory. Sample data qualification based on all available blank results (laboratory MB, equipment blank, and other field blanks) is performed by the user (client). The processing of blanks helps eliminate reporting of false positives in project reports.

11.5.1 Method blanks should be free of target analytes and other interfering non-target analytes. To be acceptable, blank results should not exceed one half of the applicable MQL. The default MQL is 1-mg/L for aqueous samples and 8-mg/kg for soil samples.

11.5.2 CHECK - The instrument has a calibration routine that requires the y-intercept to be updated periodically to reflect current conditions. If the y-

intercept is not updated, sample and blank results may be biased up to 0.6-mg/L. Review the results of all associated blanks and verify that the method blank has been reported for each matrix and for each system used to analyze associated samples. If levels greater than one half the PQLs are present, inspect and correct the problem. Reanalyze all associated samples. Contact client if other criteria are applicable. The only exception may be quick turn around projects.

11.5.3 ACTION – All positive results are reported with a "B" code, if contaminants are found in the method blank.

11.5.4 ACTION – If a compound is detected only in the blank and not in any of the associated samples, then no action is taken.

11.5.5 ACTION – Any compound detected in the sample which were also detected in associated method blank at levels greater than one half the PQLs should be re-extracted and/or re-analyzed unless other client criteria are applicable.

11.6 LABORATORY CONTROL STANDARD (LCS) – The LCS analysis is used to evaluate method competence in the absence of matrix effects. The LCS should be prepared from the same source as the ICAL and CCV standards. The LCS may provide a mechanism to check method efficiency for target compounds without the sample matrix as a complicating factor. Document LCS/LCSD results in Form-1 equivalents and recoveries/RPDs in Form-3 equivalents (see Appendix 8 for details).

11.6.1 CHECK - Calculate LCS recoveries and compare them with the QC limits (Tables 1 and 2) to see if the recoveries are within the acceptable range (70% to 130%). Flag outliers with an asterisk (*).

11.6.2 ACTION – Check the results for the CCV, which is virtually identical to the LCS sample. Reanalyze the LCS or check the LCS duplicate sample results. If the CCV and LCSD results and the results for the second analysis are outside the QC limits, then the LCS, method blank, and all associated samples of the batch should be reanalyzed.

11.6.3 ACTION - If sufficient sample is not available for reanalysis, or if the corrective action is ineffective, discuss the situation with the client project manager (or chemist). Include a discussion of the situation in the case narrative. The case narrative should discuss the corrective action taken and any other information. Professional judgment may be used by the user in evaluating sample data qualification options in conjunction with other QC data for the project.

11.7 MATRIX SPIKE/MATRIX SPIKE DUPLICATES – Matrix spike data are used to evaluate the precision and accuracy of the analytical method on project samples. Document MS/MSD results in Form-1 equivalents and recoveries/RPDs in Form-3 equivalents (see Appendix 5 for details).

11.7.1 CHECK - Calculate MS and MSD recoveries and compare them with the QC limits (tables 1 and 2) to see if the recoveries are within the acceptable range (70% to 130%). Flag outliers with an asterisk (*).

11.7.2 CHECK - Calculate relative percent difference (RPD) between the MS result and the MSD result. Do not use recoveries to calculate RPDs. Compare the observed RPD with the QC limit (30%) to see if the RPD is within the acceptable range (30%). Flag outliers with an asterisk (*).

11.7.3 ACTION – MS data evaluation is more complex than method blank or LCS data evaluation since they are complicated by matrix effects in addition to sample preparation and analysis errors. The relatively small size (1o to 100-mg) of the grab soil and water (0.5-mL) samples further complicate the evaluation since it is assumed that the native concentrations are constant in triplicate analyses. In addition, concentrations of the target analytes in the sample can also far exceed spike amounts.

11.7.4 ACTION - If the native concentration of target analytes in the sample chosen for spiking is high relative to the spiking concentration, the differences in the native concentration between the unspiked sample and the spiked samples may also become very significant.

11.7.5 ACTION - If the sample chosen for MS/MSD is inappropriate, notify the client. When additional payments are authorized for re-analysis, re-extract and/or reanalyze the MS/MSD samples using dilution techniques to minimize matrix interference, or using other project samples that appear to be clean, when sufficient quantities are available. Re-spike the sample at a higher level (e.g., at approximately two to four times the sample concentration), then reanalyze the sample based on project-specific requirements. It is the responsibility of the client at all times to select appropriate samples for MS analysis, make payments for additional analyses, and to offer necessary guidance as needed to meet their data quality objectives.

11.7.6 ACTION - No action is taken on MS/MSD recovery data alone to qualify all associated samples. A general rule is to have the recoveries within the control limits. Since the presence of significant amounts of matrix interference could be present, the supervisor should be consulted when significant outliers are noted. Document QC outliers for %recovery and RPD on the checklist and on client reports (*) and draw attention to the QC outliers on the case narrative. No action may be taken on MS/MSD recovery data alone to qualify associated samples either by the analyst or by the end user. Professional judgment may be used by the user in evaluating data qualification options in conjunction with other QC data (e.g. LCS and/or LCSD recoveries) for the project.

11.8 SAMPLE RESULTS - Results greater than the MDL (see Appendix 9 for details) or the lower level for reporting (LLR or RL) as appropriate should be reported as detected. Data qualifiers should be used to indicate values in excess of the upper calibration limit or lower than the lower calibration limit. The detection of TOC at levels greater than one half the method quantitation limit (MQL) in the associated method blank should also be indicated through appropriate qualifiers.

11.8.1 The method detection limit (MDL) for TOC and DOC analysis at the selected concentration range (0 to 20-mg/L) is 0.14-mg/L and the method reporting limit (MRL) is 0.5-mg/L. The MQL is 1-mg/L.

11.8.2 The method detection limit (MDL) for TOC and DOC analysis at the selected concentration range (0 to 20-mg/L) is 0.2-mg/kg and the method reporting limit (MRL) is 0.4-mg/kg. The MQL is 0.8-mg/kg.

11.8.3 ACTION - Report positive results that are greater than either the MDL and/or the reporting limit (as appropriate) but lower than the MQL as estimated with a "J" flag.

11.8.4 Report results greater than the upper limit of calibration with as estimated with an "E" flag. Such samples will require reanalysis with dilution.

11.8.5 Adjust sample results and associated MDL, RL, and PQL, for dilution, and non-standard sample weights for soil samples. Do not adjust for percent moisture since acid-treated and dried samples are used for analysis.

12.0 REFERENCES

12.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 9060, Total Organic Carbon, September 1996.

12.2 Environmental Protection Agency. Methods for Chemical Analysis of Water and Wastes, Organic Carbon, total, Method 415.1 (combustion or oxidation) and Method 415.2 (low level UV promoted, persulfate oxidation), EPA-600/4-79-020, March 1983.

12.3 Annual Book of ASTM Standards, Part 31, "Water" Standard D 2574-79, p 469 (1976)

12.4 Standard Methods for the Examination of Water and Wastewater, Method 5310, "Total Organic Carbon (TOC)," 20th Edition, 1998.

12.5 Apollo 9000 TOC Combustion Analyzer, User Manual, Tekmar-Dohrmann, Document part number 14-9000-074, Rev A, Version 05.21.99.

12.6 Installation and Operation of the 183 Boat sampling Module with Apollo 9000, Tekmar-Dohrmann, Document part number 14-7723-074, Rev A, April 2000.

12.7 Method detection limit (MDL) as described in "Trace Analyses for Wastewater," J. Glaser, D. Foerst, G. McKee, S. Quave, W. Budde, Environmental Science and Technology, Vol. 15, Number 12, p. 1426, December 1981.

12.8 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

12.9 AML Standard Operating Procedures, Q-Water-RC.

12.10 AML Standard Operating Procedures, Q-WasteMgmt-RD.

12.11 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

12.12 AML Standard Operating Procedures, Q-ControlCharts-RC.

Table 1
Aqueous MDLs, MQLs, and QC Limits for LCS/LCSD and MS/MSD

Total Organic Carbon (TOC) by Combustion-NDIR Spectroscopy

Analytical Management Laboratories

Matrix: Water	**Sample Preparation**				**Sample Analysis**						**Technique**			
Analyte	Method No.		Date		Method No.		Date							
Total Organic Carbon	9060		11/1/01		9060		11/1/01				NDIR Spectroscopy			
Total Organic Carbon	9060		12/12/01		9060		12/12/01				NDIR Spectroscopy			
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**		**LCS Limits**		**MS Limits**		**RPD**
Analyte	Amount	mg/L	mg/L	**mg/L**	RD=B/A	flag	**Mg/L**	RQ=C/A	flag	Lower	Upper	Lower	Upper	Limit
	mg/L	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%
Total Organic Carbon	0.50	0.320	0.045	**0.142**	2.3		1.0	7.0		70	130	70	130	30
Total Organic Carbon	0.50	0.612	0.049	**0.154**	4.0		1.0	6.5		70	130	70	130	30
TOC (for reports)				**0.15**						**70**	**130**	**70**	**130**	**30**
Mean Ratio (RD or RQ):					3.1			6.8						

Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 5.

Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.

LCS and MS %R limits are laboratory limits with guidance from AFCEE QAPP and USACE EM200-1-3, Appendix I.

Table 2
Soil MDLs, MQLs, and QC Limits for LCS/LCSD and MS/MSD

Total Organic Carbon (TOC) by Combustion-NDIR Spectroscopy														
Analytical Management Laboratories														
Matrix: Soil	**Sample Preparation**				**Sample Analysis**						**Technique**			
Analyte	Method No.				Method No.									
Total Organic Carbon	9060				9060						NDIR Spectroscopy			
	Spike	Mean	S.D.	**MDL**	Ratio (RD)		**MQL**	**Ratio (RQ)**		**LCS Limits**		**MS Limits**		**RPD**
Analyte	Amount	mg/kg	mg/kg	**mg/kg**	RD=B/A	flag	**Mg/kg**	RQ=C/A	flag	Lower	Upper	Lower	Upper	Limit
	mg/kg	B		**A**			**C**			**%R**	**%R**	**%R**	**%R**	%
Total Organic Carbon	4.0			**1.2**			8.0	6.7		70	130	70	130	30
Mean Ratio (RD or RQ):														
Water MDL data was used to estimate soil MDLs using dilution factor (8) for soil.														
Flags (*) are used for mean/MDL ratios (RD) that are less than 1 or greater than 5.														
Flags (*) are used for MQL/MDL ratios (RQ) that are less than 3.														
LCS and MS %R limits are laboratory limits with guidance from AFCEE QAPP and USACE EM200-1-3, Appendix I.														

Table 3
List of Appendices

Appendix Number	Appendix Topic
1	Instrument Printouts Initial Calibration (ICAL) Summary (Form-6 equivalents) Linear Regression Parameters Calibration Verification (ICV and CCV) Summary (Form-7 equivalents)
2	Excel Spreadsheets for Verification of Instrument Calculations
3	Summary of Instrument Operation
4	QC Association Forms (Form-4 equivalents) Sample Preparation Logsheets Instrument Run Log
5	MS and MSD Sample Analysis Reports (Form-1 equivalents) MS/MSD %R Summaries and RPD Reports (Form-3 equivalents)
6	Checklist for Review and Data Verification
7	Sample Reports (Form-1 equivalents)
8	LCS/LCSD Sample Analysis Reports (Form-1 equivalents) LCS/LCSD %R Summaries and RPD Reports (Form-3 equivalents)
9	Method Detection Limit (MDL) Study Summary
10	USACE Method Quality Objectives for Method 8081A AFCEE MQOs for Method 8081A

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The forms cited in the table are EPA contract laboratory program (CLP) forms that are commonly used in CLP method documents. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices.

STANDARD OPERATING PROCEDURES

No. I-Solids-160_3-RC

Residue, Total by Gravimetry (EPA 160.3 and SM 2540B)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Total Solids or Total Residue
(METHOD 160.3 Gravimetric, Dried at 103-105 ^{o}C)

1.0 Scope and Application

1.0 This SOP is based on EPA Method 160.3 and Standard Method 2540B for the determination of total residue (total solids) by gravimetry. This method is applicable to drinking, surface and saline waters, domestic and industrial wastes.

1.1 The practical range of the determination is 10 mg/L to 20,000 mg/L.

2.0 Summary of Method

2.1 A well-mixed aliquot of the sample is quantitatively transferred to a pre-weighed Erlenmeyer flask and evaporated to dryness at 103-105 ^{o}C.

3.0 Definitions

3.1 Total Residue is defined as he sum of the homogenous suspended and dissolved materials in a sample.

4.0 Sample Handling and Preservation

4.1 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4 ^{o}C, to minimize microbiological decomposition of solids, is recommended.

5.0 Interferences

5.1 Non-representative particulates such as leaves, sticks, fish and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result.

5.2 Floating Oil and Grease, if present, should be included in the sample and dispersed by a blender device before aliquoting.

6.0 Apparatus

6.1 Erlenmeyer Flasks, 125mL

6.2 Drying oven, 103-105°C

6.3 Desiccator

6.4 Analytical balance, capable of weighing to 0.1 mg

7.0 Procedure

7.1 Heat the clean Erlenmeyer Flask to 103-105 ^{o}C for one hour. Remove to desiccator and store until needed. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg). Weigh immediately before use. After weighing, handle the flask with forceps or tongs only.

7.2 Transfer a measured aliquot of sample to the pre-weighed dish and evaporate to dryness in a drying oven.

7.2.1 Choose an aliquot of sample sufficient to contain a residue of at least 20-mg. To obtain a weighable residue, successive aliquots of sample may be added to the same dish.

7.2.2 If evaporation is performed in a drying oven, the temperature should be lowered to approximately 98 ^{o}C to prevent boiling and splattering of the sample.

7.3 Dry the evaporated sample for at least one hour at 103-105 ^{o}C . Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained or until weight loss is less than 0.5 mg.

8.0 Calculation

8.1 Calculate TSS as follows:

$$\text{TSS, mg/L} = \frac{(A - B) * 1000}{C}$$

where:

A = weight of sample + dish in g
B = weight of dish in g
C = volume of sample used in Liters

9.0 References

9.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Residue, Total, Method 160.3, Gravimetric, Issued 1971.
9.2 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Total Solids, Method 2540E.
9.3 AML Standard Operating Procedures, Q-Water-RC.
9.4 AML Standard Operating Procedures, Q-WasteMgmt-RD.
9.5 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
9.6 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. I-TSS-160_2-RC

Total Suspended Solids
(EPA 160.2)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Total Suspended Solids (EPA METHOD 160.2)
Non-Filterable Residue

1.0 Scope and Application

1.1 This SOP for the determination of total suspended solids or non-filterable residue is based on EPA Method 160.2. This SOP is applicable to drinking, surface and saline waters, domestic and industrial wastes.
1.2 The practical range of the determination is 4 mg/L to 20,000 mg/L.

2.0 Summary of Method

2.1 A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 103-105 ^{o}C.
2.2 The filtrate from that method may be used for Total Dissolved Solids (TDS or Residue, Filterable).

3.0 Definitions

3.1 Residue, Non-filterable, is defined as those solids which are retained by a glass fiber filter and dried to constant weight at 103-105 ^{o}C.

4.0 Sample Handling and Preservation

4.1 Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result.
4.2 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4 ^{o}C, to minimize microbiological decomposition of solids, is recommended.

5.0 Interferences

5.1 Filtration apparatus, filter material, pre-washing, post-washing and drying temperature are specified because these variables have been shown to affect the results.
5.2 Samples high in Dissolved Solids (Filterable Residue) such as saline waters, brines and some wastes, may be subject to a positive interference. Care must be taken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter (7.5) minimizes the potential interference.

6.0 Apparatus

6.1 Glass fiber filter discs, 4.7cm or 2.1 cm, without organic binder, Reeve Angel type 934-AH, Gelman type A/E or equivalent. NOTE: Because of the physical nature of glass fiber filters, the absolute poor size cannot be controlled or measured. Terms such as "pore size", collection efficiencies and effective retention are used to define this property in glass fiber filters. Values for these parameters vary for the filters listed above.

6.2 Filter support: filtering apparatus with reservoir and coarse (40-60 microns) fritted disc as a filter support. NOTE: Many funnel designs are available in glass or porcelain. Some of the most common are Hirsch or Buchner funnels, membrane filter holders and Gooch crucibles. All are available with coarse fritted disc.

6.3 Suction flask, 500 mL.

6.4 Gooch crucibles, 25mL (if 2.1 cm filter is used).

6.5 Drying oven, 103-105°C

6.6 Desiccator

6.7 Analytical balance, capable of weighing to 0.1 mg

7.0 Procedure

7.1 Preparation of glass fiber filter disc: Place the disc on the membrane filter apparatus or insert into bottom of suitable Gooch crucible with wrinkled surface up. While vacuum is applied, wash the disc with three successive 20-mL volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove both crucible and filter and dry in an oven at 103-105 ^{o}C for one hour. Remove to desiccator and store until needed. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg). Weigh immediately before use. After weighing, handle the crucible/filter with forceps or tongs only.

7.2 Selection of Sample Volume

For a 4.7-cm diameter filter, filter 100 mL of sample. If weight of captured residue is less than 1.0 mg, the sample volume must be increased to provide at least 1.0 mg of residue. If other filter diameters are used, start with a sample volume equal to 7 mg/cm^2 of filter are and collect at least a weight of residue proportional to the 1.0 mg stated above.

NOTE: If during filtration of this initial volume the filtration rate drops rapidly, or if filtration times exceeds 5 to 10 minutes, the following scheme is recommended: Use an unweighed glass fiber filter of choice affixed in the filter assembly. Add a known volume of sample to the filter funnel and record the time elapsed after selected volumes have passed through the filter. Twenty-five mL increments for timing are suggested. Continue to record the time and volume increments until filtration rate drops rapidly. Add additional sample if the filter funnel volume is inadequate to reach a reduced rate. Plat the observed time verses volume filtered. Select the proper

filtration volume as that just short of the time a significant change in filtration rate occurred.

7.3 Assemble the filtering apparatus and begin suction. Wet the filter with a small volume of distilled water to seat it against the fritted support.

7.4 Shake the sample vigorously and rapidly transfer 100 mL (or predetermined volume selected (7.2)) to the funnel by means of a 100 mL graduated cylinder. Remove all traces of water by continuing to apply vacuum after sample has passed through.

7.5 With suction on, wash the graduated cylinder, filter, non-filterable residue and filter funnel wall with three portions of distilled water allowing complete drainage between washing. Remove all traces of water by continuing to apply vacuum after water has passed through. NOTE: Total volume of wash water used should equal approximately 2 mL per cm^2. For a 4.7-cm filter the total volume is 30 mL.

7.6 Carefully remove the crucible and filter from the crucible adapter. Dry at least one hour at 103-105 ^oC . Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained or until weight loss is less than 0.5 mg.

8.0 Calculation

8.1 Calculate TSS as follows:

$$\text{TSS, mg/L} = \frac{(A - B) * 1000}{C}$$

where:

A = weight of crucible/filter + residue in g
B = weight of crucible/filter in g
C = volume of sample used in Liters

9.0 References

9.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Non-Filterable Residue, Total, Method 160.2.

9.2 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Total Suspended Solids.

9.3 AML Standard Operating Procedures, Q-Water-RC.

9.4 AML Standard Operating Procedures, Q-WasteMgmt-RD.

9.5 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.

9.6 AML Standard Operating Procedures, Q-ControlCharts-RC.

STANDARD OPERATING PROCEDURES

No. W-Flashpoint-CC-RC

IGNITABILITY - FLASHPOINT DETERMINATION BY CLOSED-CUP TESTER (SW-846 Method 1020B)

October 28, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

IGNITABILITY - FLASHPOINT DETERMINATION BY CLOSED-CUP TESTER (SW-846 Method 1020B)

1.0 SCOPE AND APPLICATION

This standard operating procedure is based on SW-846 Method 1020A, 1010, ASTM D-3278-78, Test Method for Flash Point of Liquids by Setaflash Closed Cup Tester and ASTM D 93-80, Test Methods for Flash Point by Pensky-Martens Closed Tester. These methods use closed-cup testers to determine the flash point of solids and liquids including those that tend to form a surface film under test conditions. Liquids containing non-filterable, suspended solids will also be tested using this SOP. The procedure may be used to determine whether a material will or will not flash at a specified temperature (e.g. room temperature) or to determine the finite temperature at which a material will flash up to 100°C.

2.0 SUMMARY OF METHOD

The sample is heated at a slow, constant rate and a small flame is directed into the cup at regular intervals. The flash point is the lowest temperature at which application of the test flame ignites the vapor above the sample.

3.0 SAMPLE HANDLING AND PRESERVATION

Samples will be stored at 4°C in a closed jar. Samples with flashpoint lower than room temperature will be loaded directly into sample cups without allowing them to equilibrate to room temperature.

4.0 INTERFERENCES

Liquids that tend to form a surface film may yield erroneous results with Setaflash testers. These samples will be tested with Pensky-Martens Closed Cup Testers.

5.0 APPARATUS

Koehler Model K-14600 closed cup tester.

6.0 REAGENTS

Diisobutylketone (DIBK) and p-Xylene. Prepare a standard containing DIBK and p-Xylene (75:25 v/v).

7.0 PROCEDURES

Verify that the thermometers that read sample and bath temperatures are working correctly. Verify that the heating bath contains sufficient water to keep the sample cup completed surrounded by the bath. Open the sample cup and introduce 2 ml of the

standard. Connect the gas line to the tester and light the flame. Raise the temperature of the bath at the rate of 10°F /min. until the bath temperature is approximately 5°F below the specification temperature (102°F for the DIBK/Xylene mixture or 79°F for p-Xylene). Lower the rate of heating by gently adjusting the rate of heating to approx. 4°F/min. Apply the test flame by turning the knob towards you and note whether the sample flashes or not at 15 second intervals. Record the minimum temperature at which the sample flashes. The tester is functioning correctly, if the observed flashpoint is within the limits shown below:

Standard	Expected flashpoint (°F)	Limits
DIBK/p-Xylene (75:25 v/v)	104	99 to 109°F
p-Xylene	79	76 to 83°F

Proceed with unknown sample analysis if the observed flashpoint is within the limits shown above. Otherwise, check the accuracy of the thermometers, integrity of the standards, etc. Take appropriate corrective action to restore tester to proper operating conditions.

Introduce sufficient sample (approx. 5 g or 5 ml) directly into a clean sample cup, which has been brought to either room temperature or below the expected flash point, whichever is lower. The temperature of the apparatus is raised at the rate of 10°F/min. by appropriate adjustment of the temperature dial. After each minute, a test flame is applied inside the cup and note is taken as to whether the test sample flashes or not. If the sample flashes, record the temperature range for the flash and cool the cup to approximately 10° below the flash temperature. Discard the sample and load the cup with fresh sample. Raise the temperature at the rate of 4°F/min testing for flash every 15 seconds. Record the actual flash temperature to an accuracy of 1°F. If a repeat test is necessary, a fresh sample should be used. Stop flashpoint measurements at 210°F.

8.0 CALCULATIONS

Calculations are not required for this physical measurement. Report the flashpoint in°F. If the sample flashes at room temperature, report the flashpoint as <RT°F, where RT is the actual numerical value of the observed flash temperature. If the sample does not flash at 210°F, report the flashpoint as >210°F.

9.0 QUALITY CONTROL

Sample cups must be kept clean, making sure that they do not contain flammable solvents. If solvents are used to clean the sample cup, it must be dried thoroughly prior to use. A minimum of one control standard should be analyzed with each batch of 20 samples. Samples that flash in the 135 to 145°F ranges should be reanalyzed to determine the precision and accuracy of the measurement.

10.0 REFERENCES

10.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 6, Properties, December 1996.

10.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 7, section 7.2.2(1) (a), Characteristics Introduction and Regulatory Definitions, December 1996.

10.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 1020A, Closed-cup Method for Determining Ignitability.

10.4 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 1020B, Small Scale Closed-cup Method for Determining Ignitability, Update IIIB.

10.5 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001. Appendix I.

10.6 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

10.7 AML Standard Operating Procedures, Q-Water-RC.

10.8 AML Standard Operating Procedures, Q-WasteMgmt-RD.

STANDARD OPERATING PROCEDURES

No. I-VolatileSolids-160_4-RC

Volatile Solids
(EPA 160.4 and SM 2540E)

November 2, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Volatile Solids (Method 160.4, Gravimetric, Ignition at 550°C)

1.0 Scope and Application

1.1 This SOP is based on EPA Method 160.4 and Standard Method 2540Efor the determination of volatile solids by gravimetry.
1.2 The test is useful in obtaining a rough approximation of the amount of organic matter present in the solid fraction of sewage, activated sludge, industrial wastes, or bottom sediments.

2.0 Summary of Method

2.1 A well-mixed sample is filtered through a standard glass fiber filter for the determination of total, filterable, or non-filterable residue. The residue obtained is ignited at 550 °C in a muffle furnace. The loss of weight on ignition is reported as mg/L volatile residue.

3.0 Comments

3.1 The test is subject to many errors due to the loss of water of crystallization, loss of volatile organic matter prior to combustion, incomplete oxidation of certain complex organics, and decomposition of mineral salts during combustion. The results should not be considered an accurate measure of organic carbon in the sample, but may be useful in the control of plant operations. The principal source of error in the determination is failure to obtain a representative sample.

4.0 Sample Handling and Preservation

4.1 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4 °C, to minimize microbiological decomposition of solids, is recommended.

5.0 Interferences

5.1 See comments above.

6.0 Apparatus

6.1 Glass fiber filter discs, 4.7cm or 2.1 cm, without organic binder, Reeve Angel type 934-AH, Gelman type A/E or equivalent.
6.2 Filter holder, membrane filter funnel or Gooch crucible adapter.
6.3 Suction flask, 500 mL.
6.4 Gooch crucibles, 25mL (if 2.1 cm filter is used).
6.5 Ceramic crucibles
6.6 Muffle furnace, 550°C ± 10°C

6.7 Desiccator
6.8 Analytical balance, capable of weighing to 0.1 mg

7.0 Procedure

7.1 Preparation of glass fiber filter disc: Place the disc on the membrane filter apparatus or insert into bottom of suitable Gooch crucible. While vacuum is applied, wash the disc with three successive 209-mL volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Discard washings.
7.2 Preparation of Ceramic crucibles: Heat clean crucibles to 550 °C ± 10 °C for one hour. Cool in desiccator and store until needed. Weigh immediately before use.
7.3 Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 100 mL to the funnel by means of a 100-mL graduated cylinder. If total filterable residue is low, a larger volume may be filtered.
7.4 Filter the sample through the glass fiber filter, rinse the three 10 mL portions of distilled water and continue to apply vacuum for about 3 minutes after filtration is complete to remove as much water as possible.
7.5 Transfer the filter paper containing the residue to a pre-weighed ceramic crucible. Heat it for at least one hour at 550 °C ± 10 °C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained or until weight loss is less than 0.5 mg.

8.0 Calculation

8.1 Calculate VS as follows:

$$\text{VS, mg/L} = \frac{(A - B) * 1000}{C}$$

where:
A = weight of combustion residue + dish in g
B = weight of dish in g
C = volume of water sample used in Liters

9.0 References

9.1 Methods for chemical analysis of water and wastes, EPA-600/4-79-020, March 1983, Volatile Residue, Method 160, Gravimetric, Issued 1971.
9.2 Standard methods for the Examination of Water and Wastewater, 20th Edition, 1998, Fixed and Volatile Solids Ignited at 550°C, Method 2540E.
9.3 AML Standard Operating Procedures, Q-Water-RC.
9.4 AML Standard Operating Procedures, Q-WasteMgmt-RD.
9.5 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
9.6 AML Standard Operating Procedures, Q-ControlCharts-RC.

APPENDIX TO STANDARD OPERATING PROCEDURES

No. Q-Appendix-ManualIntegration-RC

MANUAL INTEGRATION
AML POLICY AND PROCEDURES

October 31, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Table 1
List of Available Appendices

Appendix Number	Appendix Topic
1	USEPA Region V Central Regional Laboratory, Chicago, IL SOP # HK014 on Chromatographic Peak Integration Procedures
2	Figures from Appendix A of the USEPA Region V CRL SOP # HK014 Proper Integration Procedures
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APPENDIX 1

USEPA Region V Central Regional Laboratory, Chicago, IL SOP # HK014 on Chromatographic Peak Integration Procedures

APPENDIX 2

Figures from Appendix A of the USEPA Region V CRL SOP # HK014 Proper Integration Procedures

APPENDIX 3

Figures from Appendix B of the USEPA Region V CRL SOP # HK014 Improper Integration Procedures

APPENDIX 4

Figures from Appendix C of the USEPA Region V CRL SOP # HK014 Situations Requiring Analyst Intervention for Peak Integration

STANDARD OPERATING PROCEDURES

No. Q-Audit-RE

INTERNAL QUALITY ASSURANCE AUDITS AND PERFORMANCE EVALUATION

October 31, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This Analytical Management Laboratories (AML) standard operating procedure (SOP) provides general information on quality assurance (QA) audit procedures and guidance for conducting internal QA audits. This SOP also provides guidance to QA personnel for laboratory certification, technical performance evaluation of laboratory personnel and the laboratory.

1.2 Successful and effective implementation of the AML QA Program (QAP) requires development, routine checks and periodic assessment of the degree of its implementation and the effectiveness of the program in controlling quality of the analytical process. Periodic assessment is performed by conducting audits to review AML procedures and to verify that they meet the goals and objectives of the QAP.

1.3 Audits can be internal or external. Results from the external and internal audits are used to continually monitor lab performance and correct any out-of-control situations. Overall, the audit system is expected to identify strengths and more importantly areas of weakness so that appropriate corrective actions may be taken to alleviate problems.

1.4 Other objectives of the audit procedure include the following: to facilitate the initiation of changes aimed at enhancing the QAP; to serve as a vehicle for providing technical assistance and/or training, as appropriate; to enhance awareness and understanding of quality control (QC) and QA policies and procedures; and to provide a measurement of the effectiveness of QC in assuring the quality of data.

2.0 SUMMARY OF THE QA AUDIT PROCEDURES

2.1 Internal performance audits will be planned and executed by the Quality Assurance Director (QAD) at least quarterly. The quarterly audits, which will rotate through various technical areas, may not cover all technical areas each quarter. On an annual basis, all technical areas will be covered.

2.2 These audits will consist of a technical audit of the written standard operating procedures (SOP), deviations of the procedures from the EPA methods on which they are based, actual procedures followed.

2.3 The audits may involve the analysis of a combination of blind performance evaluation (PE) audit samples, Standard Reference Materials (SRMs), known standards, or blind spike samples.

2.4 Upon completion of the audits, a report containing observed deviations, reported results for PE samples, if any, true values and control limits will be sent to the Team Leaders and the Laboratory Director. The report will also outline areas of concern and it will prompt review and implementation of appropriate corrective action to alleviate problem areas. The report and any response received from the Team Leaders will become a part of the QA reports to the management.

3.0 INTERFERENCES
Not Applicable

4.0 APPARATUS AND MATERIALS
Audit Checklist

5.0 REAGENTS
Performance evaluation (PE) samples (APG, ERA, etc.). PE audit samples should be prepared, preserved and handled as described by the vendor in their instruction manual.

6.0 STANDARDS
Not Applicable.

7.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING
Not applicable. See Section 5.o for handling PE samples.

8.0 HEALTH AND SAFETY
Not Applicable.

9.0 WASTE DISPOSAL
Not Applicable.

10.0 PROCEDURE

10.1 **External Audits:** External QA audits are performed annually or at a frequency chosen by the external auditors and/or certifying agencies. External QA audits, which may include a site visit, may involve one or more of the following: review of laboratory quality systems, laboratory capabilities, SOPs, and PE sample results.

10.1.1 AML either participates or in the process of getting ready to participate in external audit programs offered by EPA and other agencies: National Environmental Laboratory Accreditation Program (NELAP), which replaces the EPA Water Pollution (WP) Audit Program and the U.S. Army Corps of Engineers (USACE) laboratory validation program.

10.1.2 The QA management staff (QAS), which may include the QA Director (QAD) and/or other internal or external staff including the Operations Manager (OM) will be the focal point for external audits. The QAS is responsible for planning and coordinating such activities with external agencies.

10.1.3 The QAS, Operations Manager, Team Leaders and the Laboratory Director will review PE results, audit reports and all relevant documentation for deficiencies or inconsistencies. After every performance audit, the QAS will issue a report to the certifying agency along with a copy to the Laboratory Director, outlining the corrective action taken for any unacceptable results.

10.2 **PE Samples:** If PE audit samples are used either in the external or internal audit program they are treated as actual samples and are logged in the same manner as client projects. External PE audit samples may also be used to conduct internal audits and they may be supplemented with additional PE audit samples that are different from the PE audit samples used by external agencies. Results of external PE audit samples must be reviewed by QA staff before they can be reported to accrediting agencies. The PE sample results will be compiled and maintained by the QA office and results made available to Operations Manager to make quality improvements. Performance audits should cover AML's service areas.

10.3 **Laboratory Certification**: Original certificates and copies by external agencies should be maintained by the QA office.

10.4 **Internal Technical Audits**: Internal technical audits are conducted quarterly to assess laboratory performance in stated service areas. The purpose of the internal audit is to identify strengths and weaknesses and to address weakness in order to improve laboratory performance. These quarterly audits may cover one or more technical areas. Not all areas may be covered in quarterly audits. The audit should cover the following: initial demonstration of performance for new testing services, method detection limits, performance data for laboratory control samples (when applicable), existence of written procedures that could be in the form of methods and/or SOPs, and adherence to such procedures. See Section 13 for QA SOPs that may be used as the basis of internal audits. The audit will determine whether quality control standards such as blanks, LCS, matrix spikes, duplicates, etc., are incorporated with sample analytical runs at the proper frequency. The internal audit should be conducted using AML forms shown in Appendix 1. These are generic forms should be supplemented with method specific checklists for more detailed auditing. An example method specific checklist for the explosives analysis is also included in Appendix 1.

10.5 **Internal System Audits:** System audits are performed to determine if the entire operation is performing in accordance with the policies of AML. This audit will include verification of the adherence to the QA Plan, Chemical Hygiene Plan, and QA SOPs (See Section 13) that govern ethics, project management, training, etc. that are integral parts of the operation. System audits can be internal or external. Internal system audits will be planned and executed either by AML QA Staff or by an audit team, which may include external staff at least once a year. The overall laboratory QA/QC program will be monitored at this time. Appendix 2 contains an example checklist that may be used to audit major areas of the laboratory. The system audit should involve extensive interviews of the analysts with the objective of improving and implementing more effective procedures, evaluate training needs, and to address resource requirements. At the conclusion of the audit, the QA Staff will provide a written report to the Team Leaders along with a copy to the Laboratory Director and the Operations Manager for consideration of any recommended corrective action.

11.0 QUALITY CONTROL

11.1 The QA Director is responsible for the design, audit, and implementation of the overall QA Program.

11.2 The Operations Manager is responsible for its implementation including quality control of client data.

11.3 The Laboratory Director is responsible for the overall performance of the laboratory. The Laboratory Director may perform random audits of each area including the QA area to verify that all areas of the laboratory are functioning optimally.

12.0 DATA VERIFICATION AND VALIDATION

Not Applicable.

13.0 REFERENCES

13.1 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 2001.

13.2 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

13.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 1, Quality Control, December 1996.

13.4 AML Chemical Hygiene Plan.

13.5 AML Quality Assurance Plan.

13.6 AML Standard Operating Procedure, Q-SOPs-RC, Preparation, Distribution, and Tracking of Standard Operating Procedure (SOP) Documents.

13.7 AML Standard Operating Procedure, Q-Training-RC, Technical Training.

13.8 AML Standard Operating Procedures, Q-MDL-RC, IDL, MDL, RL, PQL, and Initial Demonstration of Performance.

13.9 AML Standard Operating Procedure, Q-CCGM-RC, Generation and Maintenance of Control Charts.

13.10 AML Standard Operating Procedure, Q-DMP-RC, Data and Information Storage, Retrieval, and Management Procedures.

13.11 AML Standard Operating Procedure, Q-Ethics-RB, Ethics Statement.

13.12 AML Standard Operating Procedure, Q-DVP-RC, Data Reduction, Review, Verification, and Reporting Procedures.

13.13 AML Standard Operating Procedure, Q-ProjectMgmt-RB, Project Management.

Appendix 1

INTERNAL TECHNICAL AUDIT CHECKLISTS

Appendix 2

LABORATORY SYSTEMS AUDIT CHECKLIST

LABORATORY SYSTEMS AUDIT CHECKLIST

Analytical Management Laboratories

Quality Assurance Laboratory Procedures

General QA Procedures

_____Does laboratory has a general quality assurance plan?
_____Does laboratory has written SOPs to cover routine laboratory practices?
_____Does laboratory participates in any performance audit or accreditation programs?
_____Has corrective action been documented for unsatisfactory PE performance?

Chain-of-Custody

_____Is COC present?
_____Does sample information agrees with COC?
_____Is signature and date and time of receipt on COC?

Sample Login

_____Was project logged in correctly and promptly?
_____Was all necessary login information included?
_____Was sample condition and preservation noted?
_____Were samples assigned unique numbers and were internal and client number both included on login record?
_____Were sample containers and lids both clearly labeled?

Sample Storage

_____Were samples and sample extracts properly stored during the project?
_____Is sample storage area clean and orderly?

Labware and Sample Container Preparation

_____Does lab maintains a distilled/deionized water system?
_____Was labware preparation adequate?
_____Does lab follows written SOPs for labware preparation?
_____Were sample containers supplied by the laboratory?
_____Were sample containers purchased pre-cleaned or cleaned according to applicable protocols?
_____Are sample containers sent to client pre-preserved and is this documented?

Sample Preparation

_____Do sample preparation methods follow SOPs?
_____Is sample prep area clean and free of clutter?

Solvents, Chemicals, and Standards

_____Were the standards correctly prepared?
_____Was the standards preparation correctly documented?
_____Are standards and chemicals labeled and dated correctly?

LABORATORY SYSTEMS AUDIT CHECKLIST

Analytical Management Laboratories

Equipment Calibration and Maintenance
_____Was necessary instrument calibration met?
_____Was instrument calibration documented?
_____Were calibration logbooks for thermometers, refrigerators, R.I. oils, balances, etc. complete?
_____Are instrument maintenance logs up-to-date?
_____Is routine maintenance up-to-date?
_____Was equipment in working order at the time of the analysis?
_____Is equipment adequate for the method used?

Sample Analysis
_____Did sample analyses follow the specified method?
_____Were any deviations form the specified method documented?
_____Were the sample holding times observed?

QC Sample Analysis
_____Were the required QC samples analyzed at the proper frequency?
_____Were blank samples analyzed at the proper frequency?
_____Are QC data retrievable for all analytical results?

Raw Data Storage
_____Is the raw data properly stored and easily retrieved?
_____Is the raw data legible and well preserved?

Quality Control Data
_____Were the QC objectives met for this sample?
_____Is QC data within the desired control limits?
_____Were out-of-control samples documented?
_____Was any corrective action properly documented?

Client Report
_____Did report go through the proper validation process?
_____Were all calculations correct?
_____Was the report completed on time?
_____Were units, Detection limits, etc. correctly reported?
_____Was there any client feedback on this report?

Report Storage and Archiving
_____Is raw data for this report correctly stored or archived?
_____Was the client report for this project correctly stored or archived?

LABORATORY SYSTEMS AUDIT CHECKLIST

Analytical Management Laboratories

Administration

Client Invoice

_____Was the project invoiced in a timely manner?
_____Was the invoice correct and complete?

Policies and Procedures

_____Were Team Leaders familiar with company policies?
_____Were Team Leaders familiar with company procedures?
_____Were Team Leaders familiar with company benefit plans?
_____Were Team Leaders familiar with company goals?
_____Were employees familiar with company policies and procedures?
_____Were employees familiar with company benefit plans and its goals?

Laboratory Safety

_____Does the laboratory have a chemical hygiene plan?
_____Are MSDS's available?
_____Does the laboratory have a safety designee or safety committee?
_____Have all lab employees been trained in lab safety practices?
_____Are written records kept concerning lab safety training?
_____Does the laboratory perform safety audits periodically?
_____Are fume hoods checked periodically?
_____Are there written records for fume hood checks?
_____Are eye-washes available?
_____Are fire extinguishers present?
_____Are lab coats, gloves, and eye protection utilized where appropriate?
_____Are there any obvious safety hazards?

Sample and Waste Disposal

Does the lab have a policy (written) for treatment and disposal of lab samples and waste?
Are waste materials properly secured and stored?
How are waste materials disposed?

Employee Satisfaction

Did the employees receive adequate training to perform assigned tasks?
Were sufficient resources provided to complete tasks on time?
Annual performance evaluation performed?
Does a mechanism exist for addressing employee grievances?

Client Satisfaction

_____Did the client receive the project results on time?
_____Were we responsive to the client needs?
_____Suggestions from clients to improve our services obtained?

STANDARD OPERATING PROCEDURES

No. Q-Cleaning-RD

Glassware Cleaning Procedures

October 31, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This Analytical Management Laboratories (AML) standard operating procedure (SOP) is designed to provide guidance for cleaning and storage of glassware, plasticware, and labware that are routinely used and reused in laboratory operation. This SOP is based on the EPA Region V Central Regional Laboratory SOP on this subject. AML employees should adhere to the procedures set forth in this document.

1.2 Special labware cleaning procedures that are a part of an analytical method or SOP are not covered by this document. This document does not cover sample container preparation and the preparation, if any of disposable labware.

1.3 The procedures described here (e.g. number of rinses, etc.) are subject to modification by group leaders for specific projects that may require more (e.g. low detection limit projects) or less (e.g. for accommodating expedited turnaround projects) cleaning to meet project needs.

2.0 SUMMARY OF METHOD

2.1 Laboratoryware that are reused are scrubbed and washed with soap and tapwater. After the wash, labware is rinsed dilute acids, deionized water (DI), ASTM Type I Reagent Water, organic solvents, etc. before they are allowed to dry. Procedures used depend on the type and intended use of labware.

3.0 INTERFERENCES

3.1 Common household detergents may contain high levels of phosphate, deodorants, and other organic and inorganic chemicals, which may interfere with some analyses. Such detergents should not be used. Use only detergent such as those prepared by Alconox that are intended for laboratory operations.

3.2 The paint used for markings on beakers, etc. may contain high levels of lead and other elements, which may accumulate in the detergent bath. The detergent bath should be emptied periodically and washed thoroughly to minimize high blank levels and/or interference to laboratory analyses.

4.0 APPARATUS AND MATERIALS

4.1 Rubbermaid plastic storage box (60-quart) for detergent bath.
4.2 Brush, various sizes and shapes
4.3 Glassware racks, various sizes and shapes
4.4 Strainers
4.5 Laboratory cupboards and drawers

4.6 Aprons
4.7 Face Shields

5.0 **REAGENTS**

5.1 Liquinox, liquid detergent for critical cleaning from Alconox. Prepare a 1% solution (200-ml in 20-liters) in warm water from the tap.
5.2 Deionized water (Type II) See AML SOP on reagent water (Section 13, References).
5.3 Type I Reagent water. See AML SOP on reagent water (Section 13, References).
5.4 Dilute acids (nitric, hydrochloric, sulfuric, etc. as applicable) for rinse.
5.5 Solvents (methanol, acetone, etc.) reagent grade for rinse.

6.0 **STANDARDS**

Not Applicable.

7.0 **SAMPLE COLLECTION, PRESERVATION, AND HANDLING**

Not Applicable.

8.0 **HEALTH AND SAFETY**

See AML Chemical Hygiene Plan for details. Specific guidance is provided below.

8.1 Laboratory glassware used for sample preparation may contain acids, solvents, and standards containing a number of regulated compounds. The toxicity of these chemicals present in such complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.
8.2 Acid baths used for cleaning labware used in metals analysis may be highly corrosive. Aprons, face shields, and long rubber gloves should be worn when glassware is washed using acid baths.
8.3 Glassware known to contain organic solvents should be allowed to evaporate in a hood to minimize exposure to organic vapors before they are cleaned with soap and water.
8.4 Proper sample disposal practices (see current revision of AML SOP Q-WasteMgmt-R_) should be followed to minimize exposure to toxic compounds.
8.5 Broken glassware should be discarded or fire-polished when possible to minimize cuts and other injuries.

9.0 **WASTE DISPOSAL**

See Section 13, for references to AML SOP on laboratory waste disposal.

10.0 **PROCEDURE**

The general procedure is quite simple. However, strict adherence to the procedures and documenting deviations will be necessary to minimize contamination in the laboratory and to evaluate problems with method blanks. Procedures for various areas are shown below. Contact team leader if additional guidance is required.

10.1 Biological Oxygen Demand (BOD) BOTTLES - BOD bottles, 4 liter glass beakers, stoppers, plastic caps must be soaked in the detergent bath for 30-minutes, scrubbed thoroughly for 1-minute, rinsed three times with tap water and twice with DI water. The bottles should be allowed to air dry overnight prior to use.

10.2 MINERALS AND NUTRIENTS ANALYSIS - The procedure described in Section 10.1 should be followed. The glassware should be rinsed with Type I water prior to drying.

10.3 METALS-CERAMIC WARE - If, after soaking overnight in a detergent bath, the sediment can be removed by wiping with a paper towel, the item may be placed in the soapy water tub and treated by the METALS GENERAL PROCEDURE. If, after a day of soaking, the sediment does not come off with paper towel, the Metals Group Leader should be consulted to decide on alternate procedures such as acid soaking or rinsing with organic solvents. After removing sediment, the ceramic ware is treated as described in Section 10.4.

10.4 METALS ANALYSIS GENERAL - The following procedure does not apply to disposable plasticware commonly used at AML. Other metals analysis labware are to be submerged and filled with soapy water. The following day, the labware should be rinsed with tap water, the beakers scrubbed out with a plastic sponge brush to remove all traces of baked-on sediment. Any markings on the labware should be removed at this time. Most china marker will come off with a stiff brush. Felt pen marker must be removed with methanol. After rinsing three (3) times with tap water, rinse three (3) times with DI water. Teflon vessels from the microwave digestion apparatus should not be acid soaked. Three (3) more rinsing with Type I water are needed. All plastic or rubber accessories should be removed at this point and left to dry. The glass and ceramic labware should be washed, placed in the acid bath, filled completely, submerged and left overnight. The following day, the labware is removed from the acid bath, using long, thick rubber gloves, apron, and a full face shield, transferred to a plastic bucket, and filled with DI water. The labware is rinsed thoroughly with DI water, followed by three rinsings with Type I water, and allowed to dry in a rack. After drying, the labware should be placed in the appropriate drawers.

10.5 PIPETS, METALS AND MINERALS NUTRIENTS ANALYSIS - Pipette soaking baskets are filled with pipettes to be washed. Pipettes are placed with their tips up. The baskets are submerged in enough soapy water to cover the tips. At least weekly, or more often if necessary, the baskets should be removed and placed in the rinsing station. Tap DI water is flushed through the rinsing station at

minimum 10 times. After rinsing, the pipettes are allowed to air dry. When dry they are placed in the appropriate drawers in the room where they originated. It is important that the pipettes return to the correct room.

10.6 ORGANICS ANALYSIS GENERAL - Delicate glassware (e.g. Snyder columns, receivers (10-ml), chromatography columns, 10 to 50-ml volumetric flasks, etc.) is rinsed with tap water and submerged in the detergent bath overnight. They should not be cleaned using sonicator baths. They should be scrubbed carefully and thoroughly for 1-minute, rinsed three (3) times with tap water and three (3) times with DI water. Rinse with Type I water is not required. The glassware is then dried in the oven (exception: volumetric flasks) for about 1 hour. Put all the dried glassware in the appropriate drawers.

10.7 ORGANICS ANALYSIS SPECIAL - Special equipment such as continuous liquid-liquid extractor vessels should be cleaned should be manually washed as a whole piece without disassembling. Otherwise, the washing procedure is the same as for the general organic glassware.

10.8 LABORATORY CLEANING GENERAL - Some of these functions are performed by housekeeping personnel under contract. Check with the Operations Manager the functions that need to be performed by laboratory personnel. The following procedures should be followed:

10.8.1 DAILY - Benchtops without instruments: Wipe with mild soapy water, rinse with tap water, and wipe dry using paper towel.

10.8.2 DAILY - Benchtops with instruments: Dry feather dusting (no solution must be used) (light dusting) so that it does not affect the instrument. NOTE: Prior to cleaning check with group leader to assure that it is ok to do so.

10.8.3 DAILY- Instrument Tops: Dry feather dusting.

10.8.4 DAILY - Sinks and faucets: Clean with a mild soap solution so that no residue build-up and rinse with tap or distilled water as appropriate to assure that no soap residues remain.

10.8.5 WEEKLY - Chemical and other storage cabinets inside the laboratory: Feather dust outside of the cabinets (top and glass).

10.8.6 WEEKLY - Hood Area (fume hood): Feather dust is done on bench top, inside walls and windows. NOTE: In metals lab or some other hoods it is necessary to clean using moist paper towel. But make sure it is wiped dry right away. This is necessary to remove the residue that cannot be removed by just feather dusting alone.

10.8.7 MONTHLY - Emergency Showers: Feather dusting is done on the top of shower.

10.8.8 MONTHLY - Clocks: Feather dusting

10.8.9 QUARTERLY - Refrigerators and Ovens: Feather dusting done (top and outside); then wipe with dry clean cloth or paper towel. CAUTION: Oven may be too hot for this procedure.

11.0 **QUALITY CONTROL**

11.1 Inspect glassware visually for clarity, breakage, etc. Discard unusable labware.

11.2 Order cleaning supplies and replacement glassware to maintain adequate inventory of supplies and equipment.

12.0 DATA VERIFICATION AND VALIDATION

Not Applicable.

13.0 REFERENCES

13.1 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 2001.

13.2 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

13.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 1, Quality Control, December 1996.

13.4 EPA Region V Central Regional Laboratory Standard Operating Procedure, Number CRL-SOP-HK008, Labware Cleaning SOP, December 20, 1999.

13.5 AML Chemical Hygiene Plan.

13.6 AML Standard Operating Procedures, Q-Water-RC, Reagent Water.

13.7 AML Standard Operating Procedures, Q-WasteMgmt-RC, Waste management and disposal including sample disposal.

STANDARD OPERATING PROCEDURES

No. Q-Complaint-RC

Complaint Resolution

October 31, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Complaint Resolution

1.0 SCOPE AND APPLICATION

1.1 This Analytical Management Laboratories (AML) standard operating procedure (SOP) is designed to provide guidance for handling complaints. AML management and technical staff should adhere to the procedures set forth in this document so that complaints can be addressed satisfactorily.

2.0 SUMMARY OF PROCEDURE

2.1 Complaints can arise from clients or other parties about the laboratory's activities, policies, procedures, or the integrity of data generated.

2.2 Complaints can be internal or external to the organization. Complaints, when not addressed can cause serious disruption to the organization. This SOP provides guidance to management and technical staff for addressing them.

2.3 This document describes the most common type of complaints and the party and/or procedures that is likely to be the most effective in addressing them. Legal counsel may be needed to address complaints that are not technical in nature.

3.0 INTERFERENCES

3.1 Lack of effective communication with clients and/or third parties, potential negligence of the laboratory staff and/or the client, lack of understanding of project requirements are common sources of complaints. See AML SOP on project management to minimize the source of complaints.

3.2 Complaint resolution may become a significant problem when multiple points of contact exist for the client or party that originated the complaint. Whenever possible, a single point of contact should be encouraged to keep confusion to a minimum.

3.3 Verbal client instructions may conflict with written documents or project plans. Deviations should be documented in written internal records to protect laboratory from unscrupulous clients.

4.0 APPARATUS AND MATERIALS

4.1 Personal Computers.
4.2 Laboratory Information Management System
4.3 AML Forms
4.4 Telephone
4.5 Facsimile transmission machine (FAX)
4.6 Electronic Mail (Email)
4.7 Writing pads, pen, pencil, etc.

5.0 **REAGENTS**
Not Applicable.

6.0 **STANDARDS**
Not Applicable.

7.0 **SAMPLE COLLECTION, PRESERVATION, AND HANDLING**
Not applicable.

8.0 **HEALTH AND SAFETY**
Not Applicable.

9.0 **WASTE DISPOSAL**
Not Applicable.

10.0 **PROCEDURE**

10.1 **Type of Complaints –** Complaints may originate from the following parties:

10.1.1 Employees – This may include current employees, ex-employees, or recruits. Complaint may be related to discrimination in hiring, promotion, and/or discharge. Complaints may also be related to compensation, benefits, or health/safety issues. Contact Counsel for legal help. The President of the Company initiates the request for legal issues.

10.1.2 Public – This may include adjacent businesses, residences, and other parties that do not have a client relationship with the company. Complaints may be related to laboratory emissions, waste disposal activities or employee conduct. Contact Counsel for legal help. The President of the Company initiates the request for legal issues.

10.1.3 Government – Company activities may include business financial management, compliance with general laws and regulations applicable to most businesses, and interfacing with the authorities involved in laboratory certification and/or regulation. Serious complaint may lead to criminal investigation of the company by federal, state or local authorities for potential fraud by the company, its employees or management.

10.1.3.1 If a criminal investigation is suspected, contact Counsel for legal help immediately. The President of the Company initiates the request for legal issues. Laboratory management should fully cooperate with the government within the limits provided by the Counsel.

10.1.3.2 Contact the company accountant for help in resolving financial issues such as income taxes, property taxes, etc. and for help in dealing with financial matter in general. The President of the Company initiates the request for help from the accountant.

10.1.3.3 The QA Director should deal directly with auditors and/or regulators to resolve issues that are technical (not legal) in nature.

10.1.4 Clients – These may be related to laboratory performance that may be contractual or technical in nature. The Project Manager is the principal point of contact to resolve disputes that are contractual or technical in nature. See Section 10.2 for type of specific problems and Section 10.3 for guidance on resolution. Refer legal matter to the President of the company.

10.1.5 Third Parties - These may be related to laboratory performance that may be technical in nature. The Project Manager of the affected project and/or the QA Director are the principal point of contact to resolve disputes that are technical in nature. See Section 10.2 for type of specific problems and section 10.3 for guidance on their resolution. Refer legal matter to the President of the company.

10.2 **Types of Technical Problems –** Problems related to schedule (late reports, questions or complaints on company invoice, etc.) should be handled by the Project Manager or his/her designee. Technical problems may include the following:

10.2.1 Missed technical holding times

10.2.2 Doubts on the representativeness of sample results

10.2.3 Quality of associated QC information (MB, LCS, MS, Duplicates, etc.)

10.2.4 Quality of Calibration

10.3 **Guidance for Preventing or Handling Technical Problems –** The laboratory reports should be detailed enough to include case narratives that describe problems encountered and the corrective actions taken to overcome such problems. Acceptable data for most clients normally include quality data (data that meet all applicable data quality objectives) and best available data (data that may not meet applicable data quality objectives after two attempts). Acceptable data may be rejected as unusable or used with or without qualifiers depending on the program and project requirements. The laboratory's role is to generate acceptable data. The laboratory should never agree to generate "perfect data" since it is unachievable and it may lead to the allegation of fraudulent conduct. The laboratory may provide a revised report that addresses the client complaints, whenever possible.

10.3.1 Missed holding times – Data may still be usable. The laboratory should be proactive. The client must be contacted if this potential arises. Too often, the client sets project schedule with total disregard for the laboratory's ability to meet short holding times (e.g. Chromium-VI, BOD, anions, etc.). The AML Project Manager is responsible for contacting the client and documenting this information.

10.3.2 Sample Results – The QA Director or his designee should conduct an audit to verify representativeness by checking the log-in, and sample labeling procedures.

10.3.3 QC Information - The QA Director or his designee should conduct an audit to verify reports, QC sample data, and calculations.

10.3.4 Calibration – Perfection may not be attainable. The QA Director or his designee should conduct an audit to verify calibration reports and calculations. Allegations of peak-shaving, etc. should be dealt with as serious matter requiring legal counsel since AML technical personnel have wide latitude to make professional judgment in the generation of data.

10.3.5 Complex problems – The senior management (Laboratory Director, QA Director, and the Operations Manager) staff is responsible for formulating the proper response to client complaint. A single point of contact should be designated for the client and the laboratory.

10.4 **Records Management -** The repository for records (email, fax, records of telephone conversations, revised reports, report addendum, etc.) created during complaint resolution should be preserved to assist in resolving potential legal disputes. Records that are technical in nature should be filed with the project file. Records that are contractual in nature should be filed with client folders. Records should be readily available for review. The designated point of contact for complaint resolution is responsible for records that are communication in nature. The LIMS Manager is responsible for data management in all forms (paper and electronic) during complaint resolution. See also AML SOP on project management for additional guidance.

11.0 QUALITY CONTROL

11.1 Only appropriately trained personnel can interface with outside parties for complaint resolution.

11.2 Copies of records created during complaint resolution should be kept on file.

12.0 DATA VERIFICATION AND VALIDATION

See applicable AML SOP on this subject.

13.0 REFERENCES

13.1 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 2001.

13.2 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

13.3 EM 200-1-2, Technical Project Planning (TPP) Process, US Army Corps of Engineers, August 31, 1998.

13.4 EM 200-1-6, Chemical Quality Assurance, US Army Corps of Engineers, October 10, 1997.

13.5 ER 1110-1-263, Chemical Data Quality Management for Hazardous, Toxic, Radioactive Waste Remedial Activities, US Army Corps of Engineers, April 30, 1998.

13.6 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 1, Quality Control, December 1996.

13.7 AML Standard Operating Procedures, Q-login-RF, Sample Receiving, Login, and Tracking.

13.8 AML Standard Operating Procedures, Q-ProjectMgmt-RB, Project Management.

13.9 AML Standard Operating Procedures, Q-MDL-RC, Instrument detection limits (IDL), method detection limits (MDL), reporting limits (RL), method quantitation limits (MQL), and initial demonstration of performance (IDP).

13.10 AML Standard Operating Procedures, Q-CCGM-RC, Generation and maintenance of control charts.

13.11 AML Standard Operating Procedures, Q-SOPs-RC, Preparation of standard operating procedures (SOPs).

13.12 AML Standard Operating Procedures, Q-Training-RC, Technical training.

13.13 AML Standard Operating Procedures, Q-Audit-RD, Laboratory certification and internal quality assurance audits.

13.14 AML Standard Operating Procedures, Q-DataMgmt-RC, Data and information storage and retrieval procedures.

13.15 AML Standard Operating Procedures, Q-DVP-RC, Data reduction, review, verification, and reporting procedures.

13.16 AML Standard Operating Procedures, Q-WasteMgmt-RC, Waste management and disposal including sample disposal.

STANDARD OPERATING PROCEDURES

No. Q-ControlCharts-RD

GENERATION AND MAINTENANCE OF CONTROL CHARTS

October 31, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

GENERATION AND MAINTENANCE OF CONTROL CHARTS

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) describes the procedures that are employed to generate and maintain control charts at Analytical Management Laboratories (AML). Before control charts can be used, preliminary tests must be done to determine the precision and bias associated with analytical methods to determine that the method is capable of meeting data quality objectives (DQOs) and to determine and quantify normal performance. Control charts can be used to control subsequent performance for organic and inorganic analyses. Procedures outlined in this SOP are based on information described in U.S. Army Corps of Engineers (USACE) guidance documents, Department of Defense Quality Systems Manual for Environmental Laboratories, EPA approved documents such as "Standard Methods for the Examination of Water and Wastewater, 19th edition", research articles, and SW-846 Method 8000B for organic analysis. Procedures described in this SOP are commonly employed in environmental laboratories to control data quality.

1.2 Statistical limits are used in conjunction with client specifications for method quality objectives and good laboratory operational procedures such as trained and experienced personnel, good physical facilities and equipment, certified reagents and standards, good maintenance and calibration of instruments, and knowledgeable management.

2.0 PURPOSE

2.1 Control charts based on statistics are developed to predict positive or negative trends in the analytical processes and to determine when an analysis is out of control. Three types of control charts are commonly used in laboratories: (1) A means chart for standards from laboratory control sample (LCS) recoveries, surrogate recoveries (SR), and matrix spike (MS) recoveries; (2) A means chart for background, reagent blank, preparation blank, or method blank; and (3) A range chart for replicate analyses such as the relative percent difference (RPD) for sample duplicates (LD), LCS duplicates (LCS/LCSD) and MS duplicates (MS/MSD).

2.2 The charts are essential instruments for quality control. Examples of situations that may show up in control charts are: (1) Shift in the mean - this may be caused by incorrectly prepared standards or reagents, contamination of sample, problems in instrument calibration, or analyst error; (2) Upward or downward trend of the Mean - this may be caused by deterioration of standards or reagents, increased concentration of the standard due to evaporation of solvent or a steady shift in calibration in one direction, or changes in chromatographic behavior; and (3) Increase in variability - this may be caused by poor technique of analyst or deviation from procedure.

2.3 The charts may be used to set warning limits, control limits, and marginal failure limits so that appropriate corrective actions may be taken to address out of control situations.

The charts are used at AML in conjunction with client specifications for method quality objectives in setting laboratory quality control limits.

3.0 MEANS CHARTS

3.1 Laboratory Control Samples (LCS)

EPA guidance for LCS and MS recoveries is contained in SW-846 Method 8000B, Section 8.5 and in the newer revision of this document, SW-846 Method 8000C, Section 9.5. Recommended performance criteria are discussed in Section 8.7 of the referenced EPA document.

3.1.1 The means chart for LCS is constructed from the mean and standard deviation of analyte/compound recoveries. Ideally, a chart may be prepared for each analytical method commonly performed in the laboratory. For accurate implementation, specific charts need to be performed for each combination of the following method variables: sample matrix, sample preparation procedure, instrumental analysis procedure, instrument, and the analyst. In practice, it is cumbersome to have multiple charts, one for each combination. The procedure followed at AML is aimed at maximizing applicability by minimizing variables and by grouping less significant variables.

3.1.2 The identity of the analysts will not be a variable for any of the procedures. Implementation of standard operating procedures (SOP) will minimize analyst variations. See AML SOP on method detection limits (MDL) as shown in Section 8.

3.1.3 The identity of the instruments will not be a variable if their IDLs and/or MDLs are substantially the same. Implementation of standard operating procedures (SOP) will minimize instrument variations. If MDLs are substantially different (by a factor of 2 or more), the identity of the instruments will be used as one of the variables (e.g. HP 5973 versus HP 5970 GC/MS systems).

3.1.4 The instrumental procedure will be used as one of the variables (e.g. SW-846 8310, HPLC/UV method versus, SW-846 8270, GC/MS method).

3.1.5 The sample preparation procedures that are quite similar will not be treated as variables, if the resulting MDLs are also substantially the same (e.g. SW-846 3510 and 3520). Implementation of standard operating procedures (SOP) that use consistent initial sample volumes, final extract volumes, conditions that foster comparable extraction efficiencies, etc. will minimize preparation method variations. If MDLs are substantially different (by a factor of 2 or more) and/or the preparation procedures are substantially different, the sample preparation method will be used as one of the variables. The effect of variability may also be addressed by using reliable detection limits (RDL) or lowest level for reporting (LLR) that are higher than MDLs

from alternate sample preparation and cleanup procedures. If cleanup procedures are performed routinely, they should be grouped with the sample preparation procedure. If cleanup procedures are performed randomly, they should not be included in the sample preparation procedure.

3.1.6 The matrix (soil, water, etc.) will be used as one of the variables.

3.1.7 Perform a minimum of four analyses during initial demonstration of performance. It is preferable to perform fifteen to twenty analyses using procedures that are normally used for sample analysis. LCS analysis is performed using clean matrices such as reagent water, clean laboratory sand, anhydrous sodium sulfate, etc. The LCS samples should be spiked with all the target compounds (or analyte) at a concentration close to the mid-level of the calibration curve. They should also be spiked with all the surrogates (if applicable) at concentrations that are normally used with sample analysis.

3.1.8 Calculate the mean (p) and standard deviation (s) for the number of measurements (n) made using the following equations:

$$s = \sqrt{(\Sigma (x_i - p)^2 / (n-1))}$$

$$p = (\Sigma x_i) / n$$

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3.1.9 Calculate upper (UCL) and lower (LCL) statistical control limits, which represent approximately the 99.7% confidence interval around the mean recovery from the following equations:

$$UCL = p + 3*s$$
$$LCL = p - 3*s$$

3.1.10 Calculate upper (UWL) and lower (LWL) statistical warning limits, which represent approximately the 95% confidence interval around the mean recovery from the following equations:

$$UWL = p + 2*s$$
$$LWL = p - 2*s$$

3.1.11 Calculate upper (UMFL) and lower (LMFL) statistical marginal failure or marginal exceedance (ME) limits, which represent approximately 4 standard deviations around the mean recovery from the following equations:

$$UWL = p + 4*s$$
$$LWL = p - 4*s$$

3.1.12 Statistical limits should be updated annually, preferably semi-annually, in

April and October of each year. Recommendations and/or requirements contained in the determinative methods vary considerably depending on the specific method, matrix, EPA division, client, client auditor, laboratory examiner, State Laboratory Auditor, etc. Strict and simultaneous adherence to each of these requirements is not practical due to inconsistencies in these requirements. Consistent with the Central Limit Theorem, the mean and standard deviation data should be updated using the cumulative information since the standard deviation of the mean rapidly approaches normal values as the number of available data points or sample size (n) increases. This requires tracking n, the number of data points included in the calculation of the means and standard deviations. The pooled or cumulative means (CM) and the pooled or cumulative standard deviations (CS) are calculated from individual means (p_1, p_2, etc.), standard deviations (s_1, s_2, etc.), the number of degrees of freedom (vi), which are related to the number of data points (n_1, n_2, etc.) using the following equations:

$$CS = \sqrt{((\Sigma (v_i * s_i^{\,2}) / \sum v_i)}$$

$$CM = (\Sigma p_i * n_i) / (\sum n_i)$$

$$v_i = n_i - 1$$

3.1.13 Calculate p, s, and n at suitable intervals as described in sections 3.1.1 to 3.1.8. Calculate the pooled values of p and s (CM and CS respectively) at semiannual intervals. Calculate annual pooled values by pooling values of CM and CS calculated semi-annually. Use theses annual pooled values to calculate statistical control and warning limits, which should be updated annually. The annual limits should be calculated in April, which should be used during the following year.

3.1.14 The mean and standard deviations can be thought of as describing the normal distribution. In the absence of bias, mean recoveries should be close to 100%. However, due to losses from sample preparation and cleanup procedures, differences in the volatility of individual compounds/analytes, blank contamination, integrity of calibration standards, etc., the mean recovery values can deviate significantly from the true value (100%).

3.1.15 When applicable, compare mean and standard deviations for the in-house data with method acceptance criteria contained in the referenced analytical method(s). For example, SW-846 (See Section 8.5.4.2 of Method 8000B and Section 9.5.4.2 in Method 8000C) recommends an acceptance window of Accuracy ± (2.44 * Precision) for the performance range.

3.1.16 Compare mean and standard deviations for the in-house data with method quality objectives (MQO) contained in USACE guidance documents and the limits proposed by the DoD in their Quality Systems Manual (QSM,

Appendix 2). For example, appendix I to EM 200-1-3 (USACE Shell) contains stringent method quality objectives (Appendix 1) that provides little allowance for the performance of individual analytes in multi-analyte methods. The DoD QSM Revision 2 contains 3-sigma control limits and 4-sigma ME limits that are analyte-specific. However, the DoD QSM provides more stringent criteria for the number of analytes that may deviate from the control limits, which are less tight compared to the USACE guidelines. The DoD QSM was developed with input from USACE and its development was based on NELAP Chapter 5 on Quality Systems and the USACE Shell document. The DoD QSM document was intended to replace the army, navy, and air force documents referenced in the DoD QSM. The AML environmental laboratory made an administrative decision to adopt the DoD QSM control and ME limits as the default limits for LCS recoveries. This decision was made to address serious completeness problems for the laboratory, which arose from the use of stringent USACE MQO-based limits coupled with stringent client data validation procedures. A combination of the two procedures resulted in the rejection of data that are generally considered usable with a relatively high degree of confidence by the general environmental analytical community.

3.1.17 It is preferable to have the statistical control limits (3-sigma) within USACE and/or DoD limits. Since MQO limits are significantly tighter than limits generally contained in the referenced EPA methods, the laboratory performance is considered acceptable if the statistical warning limits (2-sigma) are within MQO limits. It is preferable to have the control limits within the DoD QSM, which are based on actual laboratory data.

3.1.18 Since statistical control limits may be too wide and the statistical warning limits too tight, use DoD QSM limits to control data quality for USACE and non-USACE projects. Quality control limits based on DoD QSM are likely to be closer to the SW-846 recommended window (Accuracy ± 2.44 sigma) as described in Section 3.1.14.

3.1.19 Report DoD QSM limits (see Appendix 2) as "QC Limits" for recoveries. Calculate and report the total number of analytes for the method and the number of analytes that exceeded the DoD QSM control limits. Report the number of analytes that can exceed the control limits as described in Table D-1 of Appendix DOD-D, “LCS Control Limits”.

3.1.20 Most DoD clients require the laboratory to submit all the data collected for their project, which includes acceptable data, marginal data, and unacceptable data. Therefore, it is imperative that a LCS recovery report be submitted even if the whole batch is rejected by the laboratory due to unacceptable recoveries. The DoD clients may accept and use data for some analytes from a failed batch while rejecting data for the other. Follow the following guidelines while reporting LCS recoveries:

3.1.20.1 Report LCS recoveries that are within the DoD QSM control limits (3-sigma) without a flag and include DoD QSM control limits (3-sigma) in the report.

3.1.20.2 Report LCS recoveries that exceed the DoD QSM control limits (3-sigma) but not the DoD QSM marginal exceedence limits (4-sigma ME limits) with the "ME" flag and include the ME limits for these analytes only.

3.1.20.3 Report LCS recoveries that exceeded the ME limits with the "ME*" flag. Appropriate corrective action, which includes reanalysis and/or reextraction/reanalysis will be required for analytes with "ME*" flag. Contact the AML Project Manager to receive input from the client.

3.1.20.4 If reextraction and reanalysis is performed, data may be submitted for all the target analytes or the failed analyes only at client's request. Follow procedures outlines in Section 3.1.20.1 to 3.20.3 for reporting guidelines.

3.1.21 There may be instances when the analysis is performed for a full set of target analytes with allowance for a small number of "ME" outliers. However, the client may request reporting only a subset of analytes, which may not have an allowance for "ME" outliers. The subset of analytes may be "contaminants of concern" requiring data of the highest quality for making sound decisions. Whenever possible, request a list of the contaminants of concern (COC) for each project to avoid these problems. If a COC list is unavailable, apply the default LCS criteria.

3.2 Matrix Spike Recoveries (MS)

EPA guidance for LCS and MS recoveries is contained in SW-846 Method 8000B, Section 8.5 and Revision3 of this document (8000c, Section 9.5). Recommended performance criteria are discussed in Section 8.7 of 8000B and Section 9.5.4.2 of 8000C.

3.2.1 The procedures to be employed to generate control charts based on matrix spike recoveries are almost identical to those described for LCS. The only exception is the use of actual environmental samples instead of a controlled matrix in step 3.1.7 above.

3.2.2 Soil, sediment and waste will be considered as one matrix (soil). Aqueous samples such as groundwater, drinking water, wastewater, TCLP leachates, etc. will be considered to be one matrix (water).

3.2.3 Exclude data for samples in which recoveries cannot be calculated due to serious matrix interference. Otherwise, use all data, including data from samples that may not meet all QC requirements.

3.2.4 Guidance is unavailable for handling MS recoveries from the USACE EM 200-1-3 and the DoD QSM manual due to the complexity involved in interpreting MS recovery data. The USACE document, EM 200-1-10 states in section 12.1, "Because of the variety and complexity of environmental matrices, it is usually impractical for environmental production laboratories to establish matrix-specific limits". This document recommends an acceptance window of ±30% for instances when the LCS acceptance window is ±20% and the spike amount is at least twice as large as the native analyte.

This document also recommends the following: "The acceptance range for MS recoveries may be set equal to the acceptance range for LCS recoveries when the MS spike concentration is much higher than the native analyte concentration (e.g. by a factor of five or ten) or when it is desirable to establish a conservative (i.e., a more narrow) MS acceptance range"; and "If the matrix spike sample from the preparation batch originates from a different project site or is suspected to be of dissimilar composition from the other samples in batch, it must not be used to qualify the other field samples".

3.2.5 The EPA SW-846 Method 8000C (sections 9.5.4.1 and 9.5.4.2) does not recommend using data from spike-to-background ratios less than 5:1 due to the allowance that need to be made for error in measurement of both the background and spike concentrations. The SW-846 document (Chapter 1) does not recommend the performance of MS recovery analyses on any sample that contains more than 1000-ppm (mg/L or mg/kg) of an analyte.

3.2.6 Use procedures described in Section 3.1 for LCS to handle QC limits for MS at AML. These control limits and the associated ME limits should be treated as "advisory limits" and sound professional judgment should be exercised in qualifying or rejecting data based on MS recovery data or the lack thereof.

3.3 Duplicate Analyses

3.3.1 Calculate relative percent difference (RPD) using the following equation:

$$RPD = \frac{(R_o - R_d) * 100}{(R_o + R_d) / 2}$$

Where,
R_o is the percent recovery in the original sample;
R_d is the percent recovery in the duplicate sample.

3.3.2 Determine RPDs using one or more of the following duplicate measurements: laboratory sample duplicates; LCS/LCSD pairs; and MS/MSD pairs.

3.3.3 Use absolute values of RPD to calculate mean (p) and standard deviation for the mean (s) from multiple duplicate measurements. See equations included in section 3.1.8 to calculate the mean and standard deviations for absolute values of RPD. A minimum of ten measurements is recommended while calculating the mean. Mean RPDs should approach zero, if signs (positive and negative) are included.

3.3.4 Calculate control limits and warning limits for absolute values of RPD using the following equations:

$$\text{Control Limit} = p + (3 * s)$$
$$\text{Warning Limit} = p + (2 * s)$$

3.3.5 Since statistical control limits may be too wide and the statistical warning limits may be too tight, use the USACE limits and/or DoD QSM limits to control quality for USACE and non-USACE projects.

3.4 Blank Analyses

3.4.1 Blank results are control charted to make qualitative decisions. In this chart, the values are recorded sequentially to see if there are any trends toward high blank values, or if a single blank value stands out as being higher than normal. High blanks may indicate problems with reagents, including reagent water, problems with calibration (e.g. metals) procedures, cross-contamination from samples and/or standards, etc. One half of the applicable MQL is used as the upper limit for blank for target analytes with the exception of common laboratory contaminants.

3.4.2 Tabulate blank values in a laboratory notebook by date and type. Record method or preparation blanks separately from calibration or reagent blanks.

4.0 QUALITY CONTROL LIMITS

4.1 Samples analyzed for U.S. Army Corps of Engineer (USACE) projects may have additional requirements that are related to USACE laboratory validation (CX), USACE District requirements, government contract requirements, specific USACE project requirements or USACE Architect/Engineer (A/E) requirements. USACE-CX requirements for acceptable control limits for LCS recoveries, MS recoveries, surrogate recoveries, and RPDs for duplicates are shown in Appendix 2 (DoD QSM limits), which replaces USACE method quality objective documents (MQO) contained in Appendix 1. Information shown in these appendices has been reproduced from the DoD and USACE documents.

4.2 Method requirements should be met for data to be legally valid. Client requirements should be met for data to be acceptable to the client and client requirements may exceed method requirements. The following guidance applies for dealing with these issues:

4.2.1 The USACE requirements shown in Appendix 1 are client specifications.

4.2.2 AML has adopted DoD QSM specifications for LCS QC limits for reporting recovery data, while using AML statistical limits for internal control. The DoD QSM limits are acceptable to most DoD clients.

4.3 Methods used by the laboratory do not employ performance based measurement systems (PBMS), and client specifications for "method performance" for the required EPA SW-846 methods may be unattainable in routine sample analysis. See Section 7.4 for guidance on implementation.

5.0 INSTRUCTIONS FOR CONTROL CHARTING

5.1 Use Excel spreadsheets and LIMS data to calculate mean and standard deviations

for LCS recoveries, MS recoveries, LCS RPDs, MS RPDs, and RPDs for sample and/or MS duplicates for each test method. Employees should contact the Laboratory Manager and/or the QA manager for specific instructions, examples, software diskettes, etc.

5.2 Copies of the complete SOPs including all applicable appendices will be made available to on-site auditors for their review and verification.

6.0 CONTROL CHARTS for SPECIFIC METHODS

6.1 Excel spreadsheets are used to document mean, standard deviation, statistical UCL, LCL, UWL and LWL for each test in each matrix. A summary of the QC limits used by the laboratory for VOCs analysis is shown in Appendix 3. Statistical treatment of in-house data used in establishing QC limits are also available. Hardcopies and/or electronic versions of these documents cannot be released to outside parties without approval by one of the following: Laboratory Manager, Laboratory Director, QA Director or the QA Manager.

6.2 Complete copies of the SOPs including all applicable appendices will be made available to on-site auditors for their review and verification.

7.0 IMPLEMENTATION

7.1 Team leaders are responsible for gathering and tabulating information necessary to calculate the necessary parameters for tests in their technical area.

7.2 The QA Director and the Operations Manager are responsible for performing the necessary calculations and for making policy decisions related to corrective action procedures that need to be implemented for each parameter during day to day analysis. Corrective action procedures based on the control charts will be described in the SOP for each test method.

7.3 The QA Director is responsible for maintaining a current copy of the control charts for use with client project plan preparation and for monitoring the overall performance of the methods at the laboratory.

7.4 Control limits shown in USACE MQOs and adopted by AML for routine use are simply objectives that may not always be attainable for 100% of the target analytes by commercial laboratories operating under schedule and cost constraints.

7.5 Client input will be necessary to determine appropriate project-specific corrective action that may be necessary to complete projects to client satisfaction. To the extent possible, the analyst should keep communication alive with client project chemists to address QA/QC problems as they arise. Laboratory SOPs and quality assurance project plans (QAPPs), when available and client input should guide necessary corrective action for specific projects.

8.0 REFERENCES

8.1 EM 200-1-1, Validation of Analytical Chemistry Laboratories, US Army Corps of

Engineers. July 1, 1994.

8.2 EM 200-1-2, Technical Project Planning (TPP) Process, US Army Corps of Engineers, August 31, 1998.

8.3 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001, Appendix I.

8.4 EM 200-1-6, Chemical Quality Assurance, US Army Corps of Engineers, October 10, 1997.

8.5 EM 200-1-10, Guidance for Evaluating Performance-Based Chemical Data, 28 December 2001.

8.6 ER 1110-1-263, Chemical Data Quality Management for Hazardous, Toxic, Radioactive Waste Remedial Activities, US Army Corps of Engineers, April 30, 1998.

8.7 Department of Defense Quality Systems Manual for Environmental Laboratories, Prepared by DoD Environmental Data Quality Workgroup, Appendix DOD-D, "LCS Control Limits", Final Version 2, June 2002.

8.8 United State Navy, Installation Restoration Laboratory Quality Assurance Guide, Chapter 5, Interim Document, February 1996.

8.9 Air Force Center for Environmental Excellence, Quality Assurance Project Plan, Version 3, March 1998.

8.10 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.

8.11 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

8.12 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 1, Quality Control, December 1996.

Table 1
List of Available Appendices
(not included in this document)

Appendix Number	Appendix Topic
1	USACE Method Quality Objectives Appendix-I to USACE EM 200-1-13
2	Appendix DOD-D "LCS Control Limits", DoD QSM for Environmental Laboratories, Final Revision 2, June 2002
3	AML Statistical Limits for Soil and Water VOCs Analysis and their Relationship to the DoD QSM and USACE MQO Limits

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The appendices to this SOP are dynamic laboratory tools that may be updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices. The appendices may be available in hardcopy form or as portable document electronic files (pdf).

STANDARD OPERATING PROCEDURES

No. Q-DataMgmt-RE

DATA STORAGE, RETRIEVAL, AND MANAGEMENT PROCEDURES

October 31, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) is intended to provide guidelines for storage and retrieval of laboratory reports and associated raw analytical data generated by Analytical Management Laboratories (AML). This SOP shall establish the minimum standards for temporary and permanent storage of analytical level II and III data. Project-specific requirements will supersede guidelines included in this SOP. This SOP shall be used in conjunction with management directives, if any, to address changes that may be necessary for rapid compliance with project requirements. Changes that result from technological advances will also be incorporated through management directives.

2.0 SUMMARY OF THE PROCEDURE

Instrument calibration and raw sample data collected in each section shall remain in bound logbooks, electronic files, binders, etc. maintained in each section. Copies of relevant data for each laboratory project or sample delivery group (SDG) are made for review, report preparation and SDG or case file archival for each AML project. Relevant data include batch-specific QC/QA summaries and sample-specific raw data. These are maintained in data folders along with final reports.

3.0 DATA MAINTAINED IN SECTIONS

3.1 The GC/MS section will review, verify accuracy and compliance and maintain the following records in bound notebooks or three-ring binders as appropriate:

- 3.1.1 Instrument Maintenance Log for each instrument. This log will include the routine preventive maintenance schedule (septum change, column clipping, injector liner change, pump oil change, etc.) and a chronological records of corrective maintenance.
- 3.1.2 Sample Analysis or Injection Log for each instrument. This log will include date, analyst initials, standard or sample numbers, computer file numbers and entries for sample dilutions, if any.
- 3.1.3 BFB (Form 5A or equivalent) or DFTPP (Form 5B or equivalent) instrument performance check and Tune Summary. This will include at a minimum the date, analyst initials, instrument ID, ion abundance criteria, observed relative abundance and the pass/fail notation. Associated sample/standard info. Need not be generated unless required for the specific project. The summary shall include a printout of the RICs and the spectra. Maintain a separate three-ring binder for each instrument to store this data in a chronological order.
- 3.1.4 Initial Calibration Summary (Forms 6A, 6B and 6C or equivalent) for each matrix (soil, water, etc.). This will include at a minimum the date, analyst initials, GC column information, matrix and instrument ID. The summary shall include a printout of the RICs and Quan Reports for all standards. Maintain a three-ring binder for each instrument in a chronological order.

3.1.5 Continuing Calibration Summary (Forms 7A, 7B and 7C or equivalent) for each matrix (soil, water, etc.). This will include at a minimum the date/time, analyst initials, GC column information, date(s) of associated initial calibration, matrix and instrument ID. The summary shall include a printout of the RICs and Quan Reports for each standard. Maintain a separate three-ring binder for each instrument to store this data in a chronological order.

3.2 The GC section will review, verify data for accuracy and compliance and maintain the following records in either bound notebooks or three-ring binders as appropriate.

3.2.1 Instrument Maintenance Log for each instrument as described in section 3.1.1.

3.2.2 Sample Analysis or Injection Log for each instrument as described in section 3.1.2.

3.2.3 Instrument Performance Evaluation Mixture (PEM) Summary. This will include a summary of the percent breakdown of endrin and the combined breakdown of endrin and DDT for the organochlorine pesticide analysis method. Data summary shall include at a minimum the date, analyst initials, GC column information and instrument ID. The summary shall include a printout of the chromatograms and Quan Reports for all standards. Maintain a three-ring binder for each instrument and each analytical method in a chronological order.

3.2.4 Initial Calibration Summary for single component analytes (Forms 6D and 6E or equivalent) for each analytical method (organochlorine pesticides, chlorophenoxy herbicides, etc.). This will include at a minimum the date, analyst initials, GC column information and instrument ID. Data summarized shall include retention times (RT) and calibration factors (CF) at five different concentrations, their mean, %RSDs, and RT windows for each compound. The summary shall include a printout of the chromatograms and Quan Reports for all standards. Maintain a three-ring binder for each instrument in a chronological order.

3.2.5 Initial Calibration Summary for multicomponent analytes (Form 6F or equivalent) for each analytical method (toxaphene, PCB Aroclors, etc.). This will include at a minimum the date, analyst initials, GC column information and instrument ID. Data summarized shall include retention times (RT) for five peaks including three required peaks and calibration factors (CF) for each peak. The summary shall include a printout of the chromatograms and Quan Reports for all standards. Include data collected as outlined in this section with similar data collected as shown in section 3.2.3 for the same analytical method.

3.2.6 Continuing Calibration Summary (Forms 7D and 7E or equivalent) for each analytical method. This will include at a minimum the date/time, analyst initials, GC column information, date(s) of associated initial calibration, instrument ID and %D for each compound or analyte. The

summary shall include a printout of the RICs and Quan Reports for each standard. Maintain a separate three-ring binder for each instrument to store this data in a chronological order.

3.3 The Metals section will review, verify data for accuracy and compliance and maintain the following records in either bound notebooks or three-ring binders as appropriate.

3.3.1 Instrument Maintenance Log for each instrument (ICP, AA or mercury analyzer) as described in section 3.1.1.

3.3.2 Sample Analysis or Injection Log for each instrument (ICP, AA or mercury analyzer) as described in section 3.1.2.

3.3.3 ICP Interelement Correction Factors (Form 11A or equivalent). This will include at a minimum the date, analyst initials, instrument ID and the correction factor for each analyte at each wavelength from high levels of Al, Ca, Fe, Mg and other metals that are likely to be present at high levels in environmental samples. The summary shall include instrument printouts for each standard that has been analyzed and associated initial and continuing calibrations, blanks, etc. Maintain a separate three-ring binder for each instrument to store this annual data in a chronological order.

3.3.4 ICP Linear Ranges (Form 12 or equivalent). This will include at a minimum the date, analyst initials, instrument ID and the maximum concentration for each analyte at each wavelength that measurements were made. The summary shall include instrument printouts for each standard that has been analyzed and associated initial and continuing calibrations, blanks, etc. Include this quarterly information in the same binder that is used to store interelement correction data archiving them in a chronological order.

3.3.5 ICP Instrumental Detection Limits (IDL, Form 10 or equivalent). This will include at a minimum the date, analyst initials, instrument ID, background level, CRDL and IDL for each analyte at each wavelength that measurements were made. The summary shall include instrument printouts for each standard that has been analyzed and associated initial and continuing calibrations, blanks, etc. Include this quarterly data in the same binder that is used to store interelement correction data archiving them in a chronological order.

3.4 The Wet Chemistry section will review, verify data for accuracy and compliance and maintain the following records in bound notebooks.

3.4.1 Instrument Maintenance Log for each instrument (e.g. spectrophotometer, pH meter, etc.) as described in section 3.1.1.

3.4.2 Sample Analysis Log for each test method or closely related tests as described in section 3.1.2.

3.4.3 All records relating to sample analysis such as weights, volumes, instrument readings for standards and samples, calibration curves, sample readings and readings for QA/QC samples.

3.5 The Sample and Waste Management section will review, verify and maintain the following records in bound notebooks and/or binders:

3.5.1 Cooler Receipt Forms.
3.5.2 Copies of chain of custody forms.
3.5.3 Sample Disposal Records.
3.5.4 In-house analysis reports on waste generated by the laboratory.
3.5.5 Waste Disposal Records. This includes copies of Waste Profile Sheets, Hazardous Waste Manifest (HWM) Forms, Land Disposal Restriction (LDR) Forms and associated paperwork.

3.6 The Quality Assurance section will review, verify and maintain the following records in bound notebooks and/or binders:

3.6.1 Laboratory Quality Assurance Manual (LQAM).
3.6.2 Standard Operating Procedures (SOP).
3.6.3 Records relating to the Initial Demonstration of Methods.
3.6.4 Method Detection Limits (MDL).
3.6.5 Control charts for accuracy and precision.
3.6.6 Statement of Qualification (SOQ).
3.6.7 Chemical Hygiene Plans and other safety related documents.

3.7 The Project and Data Management section will generate and maintain the following records in bound notebooks and/or binders:

3.7.1 A separate folder containing one copy of the report as submitted to the client. If partial reports or revised reports were submitted, a copy of each shall be included. Include facsimile cover sheets that clearly show date, time and the number of pages. Also include copies of e-mail covers, if reports were transmitted electronically. Include copies of all correspondence between laboratory personnel and the client before, during and after project execution.

3.7.2 A second folder containing one copy of all records in support of the report submitted to the client. Reference to information stored elsewhere (e.g. Volume # and page # of lab notebooks) is acceptable. Electronic records (diskettes, CD-ROMs, etc.) are also acceptable. See also section 4 of this SOP, "Data Maintained in Raw Data Folders" to determine records that shall be maintained as described in this paragraph.

3.7.3 File cabinets and/or archived boxes of data for folders described in section 3.7.1 and 3.7.2. At a minimum, records for projects that are less than one year old (from the date of sample receipt) shall be available at minimal notice.

3.7.4 A second set of filing cabinets and/or archive boxes for folders described in section 3.7.1 and 3.7.2 for projects that are more than one year old (from the date of sample receipt) but less than five years old. These folders shall be available at two day's notice.

3.7.5 A third set of archive boxes for folders described in section 3.7.1 for projects that are more than 5 but less than ten years old. Raw data folders (section 3.7.2) for projects older than 5 years old and report folders (section 3.7.1) for projects more than 10 years old shall be destroyed except for projects with written client requests for a longer record holding time. Arrangements shall be made to transfer such records to the client.

3.8 The Sales and Marketing section will maintain the following records in bound notebooks and/or binders. Note: See corporate SOPs.

3.9 The Financial Management section will maintain the following records in bound notebooks and/or binders. Note: See corporate SOPs.

3.10 The Human Resources section will maintain the following records in bound notebooks and/or binders. Note: See corporate SOPs.

4.0 DATA MAINTAINED IN RAW PROJECT DATA FOLDERS

4.1 The GC/MS section will review, verify accuracy and compliance and temporarily maintain the following records in a raw data folder for each project and provide the same to the Project and Data Management section after project completion:

4.1.1 Copies of the sample preparation logs and instrument analytical logs containing information associated with a specific AML project. Review to determine compliance with technical holding times. Prepare a note describing deviations and recommendations for flagging as appropriate.

4.1.2 Analysis Data Sheet (Forms 1A, 1B and 1C or equivalent) for each sample. This will include at a minimum the date of sample extraction, date of sample analysis, analyst initials, sample matrix, laboratory sample number and the client sample number. The data sheet shall include a list of target compounds, MDLs and/or PQLs, compound amounts, if any, units and data qualifiers. Each sample data sheet shall also include a printout of the RIC, Quan Report, raw and background-subtracted mass spectra of identified target compounds and mass spectra of all reported TICs (only if they are required) with three best library matches.

4.1.3 Internal Standard Area and RT Summary (Forms 8A, 8B and 8C or equivalent) for each AML project. This will include at a minimum the date, analyst initials, control limits for areas and RT, and the observed values for each sample.

4.1.4 Surrogate (system monitoring compounds) Recovery Summary (Forms 2A, 2B, 2C and 2D or equivalent) for each AML project. This will include at a minimum the AML project number, sample numbers and their surrogate recoveries, quality control limits for each surrogate and a flag for each surrogate compound not in compliance.

4.1.5 Method Blank Summary (Forms 4A and 4B or equivalent) for each sample extraction and/or analysis batch. Gather one or more blank summary sheet(s) for each AML project. Unless otherwise required, the

sample preparation log and the instrument analysis log will be used to document samples associated with each method blank. Each blank summary data sheet shall also include a Form 1 for the blank, a printout of the RIC, Quan Report and the raw and background-subtracted mass spectra of identified target compounds.

4.1.6 Laboratory Control Sample (LCS)/LCS Duplicate (LCSD) Recovery Summary (Forms 3A, 3B, 3C and 3D or equivalent). EPA Form 3 is generally used to record matrix spike (MS)/MS duplicate (MSD) recoveries. Form 3 needs modification (e.g. deleting the sample concentration column) to record LCS data. Gather one or more LCS recovery summary sheet(s) for each AML project. Unless otherwise required, the sample preparation log and the instrument analysis log will be used to document samples associated with each LCS/LCSD. Each LCS recovery summary data sheet shall also include Form 1, printout(s) of the RIC(s), Quan Report(s) and the raw and background-subtracted mass spectra of identified target compounds spiked into the QC sample.

4.1.7 Matrix Spike (MS)/MS Duplicate (MSD) Recovery Summary (Forms 3A, 3B, 3C and 3D or equivalent). Gather one or more MS recovery summary sheet(s) as applicable for each AML project. Unless otherwise required, the sample preparation log and the instrument analysis log will be used to document samples associated with each LCS/LCSD. Each MS recovery summary data sheet shall also include Form 1, printout(s) of the RIC(s), Quan Report(s) and the raw and background-subtracted mass spectra of identified target compounds spiked into the QC sample.

4.2 The GC section will review, verify accuracy and compliance and temporarily maintain the following records in a raw data folder for each project and provide the same to the Project and Data Management section after project completion:

4.2.1 Copies of the sample preparation logs and instrument analytical logs containing information associated with a specific AML project. Review to determine compliance with technical holding times. Prepare a note describing deviations and recommendations for flagging as appropriate.

4.2.2 Analysis Data Sheet (Forms 1D or equivalent) for each sample. This will include at a minimum the date of sample extraction, date of sample analysis, analyst initials, sample matrix, laboratory sample number and the client sample number. The data sheet shall include a list of target compounds, MDLs and/or PQLs, compound amounts, if any, units and data qualifiers. Each sample data sheet shall also include a printout of the chromatograms and a Quan Report.

4.2.3 Analytical Sequence (Form 8D or equivalent). Copies of the Sample Analysis or Instrument Injection Log or the instrument printouts are sufficient documentation unless otherwise required for specific project.

4.2.4 Surrogate (system monitoring compounds) Recovery Summary (Forms 2E and 2F or equivalent) for each AML project. This will include at a

minimum the AML project number, sample numbers and their surrogate recoveries, quality control limits for each surrogate and a flag for each surrogate compound not in compliance.

4.2.5 Method Blank Summary (Form 4C or equivalent) for each sample extraction and/or analysis batch. Gather one or more blank summary sheet(s) for each AML project. Unless otherwise required, the sample preparation log and the instrument analysis log will be used to document samples associated with each method blank. Each blank summary data sheet shall also include a Form 1, blank sample chromatogram and a Quan Report.

4.2.6 Laboratory Control Sample (LCS)/LCS Duplicate (LCSD) Recovery Summary (Forms 3E and 3F or equivalent). EPA Form 3 is generally used to record matrix spike (MS)/MS duplicate (MSD) recoveries. Form 3 needs modification to record LCS data. Gather one or more LCS recovery summary sheet(s) for each AML project. Unless otherwise required, the sample preparation log and the instrument analysis log will be used to document samples associated with each LCS/LCSD. Each LCS recovery summary data sheet shall also include a Form 1, chromatograms and Quan Reports.

4.2.7 Matrix Spike (MS)/MS Duplicate (MSD) Recovery Summary (Forms 3E and 3F or equivalent). Gather one or more MS recovery summary sheet(s) as applicable for each AML project. Unless otherwise required, the sample preparation log and the instrument analysis log will be used to document samples associated with each LCS/LCSD. Each MS recovery summary data sheet shall also include Form 1, chromatograms and Quan Reports.

4.3 The Metals section will review, verify accuracy and compliance and temporarily maintain the following records in a raw data folder for each project and provide the same to the Project and Data Management section after project completion:

4.3.1 Quality control information that is shared by multiple AML projects present problems for data storage and archival. In such cases, all the instrument printouts with the exception of sample printouts shall remain with the project with the highest need for associated raw quality control data as described below:

1. CLP (Level IV) project.
2. Level III project requiring calibration data.
3. Level III project not requiring calibration data.
4. Project with the largest number of metals analysis samples.
5. Internal AML projects.

4.3.2 The sample printouts, printouts for sample-specific QC (e.g. MS/MSD, spike samples, post-digestion spike samples and sample duplicates) and copies of sample preparation and instrumental analysis logs shall remain in the respective project folders. Reference to the project folder

containing the missing batch-specific QC information shall be placed in project folders that lack batch and instrumental QC information.

4.3.3 Level IV or CLP reports will require several qualifiers to indicate concentrations below practical quantitation levels (C qualifiers), instrumental methods used (M qualifiers) and other qualifiers (Q qualifiers) to indicate out of control or closely related QC anomalies. These qualifiers shall not be used in non-CLP reports unless specifically requested by the client in writing due to potential problems that they may create. For non-CLP reports, actual method numbers (SW-846 or other EPA method) used for instrumental analysis of sample digestates will replace method qualifiers. Methods used to prepare sample digestion (SW-846, EPA or other method numbers) will be indicated in internal reports and provided only by written request to clients requiring such information.

4.3.4 Copies of the sample preparation logs and instrument analytical logs containing information associated with a specific AML project. Review to determine compliance with technical holding times. Prepare a note describing deviations and recommendations for flagging as appropriate.

4.3.5 Analysis Data Sheet (Forms 1 or equivalent) for each sample. This will include at a minimum the date of sample digestion, date of sample analysis, analyst initials, sample matrix, laboratory sample number and the client sample number. The data sheet shall include a list of target metals, IDLs and/or reporting levels, analyte amounts, if any, units and data qualifiers. Each sample data sheet shall also include an instrument printout.

4.3.6 Initial and continuing calibration verification (Form 2A or equivalent) summary for ICP and AA. This summary shall include instrument printouts for each calibration standard that has been analyzed. The instrument printout is an acceptable summary for projects that do not require a separate summary form as long as the printout contains the following information: true value, observed value and %recovery for each analyte; initials of the analyst, date of analysis, data qualifiers and the source/number of the standards. This is a batch-specific QC element.

4.3.7 Initial and Final CRDL standard (Form 2B or equivalent) summary for ICP and AA. This summary shall include instrument printouts for each calibration standard that has been analyzed. The instrument printout is an acceptable summary for projects that do not require a separate summary form as long as the printout contains the following information: true value, observed value and %recovery for each analyte; initials of the analyst, date of analysis and the source/number of the standards. This is a batch-specific QC element.

4.3.8 Blank (Form 3 or equivalent) summary for ICP and AA. A separate Form 1 shall be prepared for the preparation blank. For initial (ICB) and continuing (CCB) calibration blanks, the instrument printout is an

acceptable summary for projects that do not require a separate summary form as long as the printout contains the following information: true value, observed value and %recovery for each analyte; initials of the analyst, date of analysis, data qualifiers and the source/number of the standards. The blank summary shall include instrument printouts for each blank that has been analyzed. This is a batch-specific QC element. This is a batch-specific QC element.

4.3.9 ICP interference check sample (Form 4 or equivalent) summary for ICP. This summary shall include instrument printouts for the check sample A (ICSA) and check sample AB (ICSAB) for all the runs associated with each check standard that has been analyzed. The instrument printout is an acceptable summary for projects that do not require a separate summary form as long as the printout contains the following information: true value, observed value and %recovery for each analyte; initials of the analyst, date of analysis and the source/number of the standards. This is a batch-specific QC element.

4.3.10 Matrix Spike (MS)/MS duplicate (MSD) or Spike Sample Recovery Summary (Form 5A or equivalent) for ICP and AA. This summary shall include instrument printouts for the spiked samples. The summary shall contain the following information: sample matrix, sample result (SR), spiked sample result (SSR), spike added (SA), % recovery (%R), control limits for %R for each analyte, initials of the analyst, data qualifiers and the date of analysis. This is a sample-specific QC element for AML data storage purposes although it may be interpreted as a batch-specific QC for data validation purposes.

4.3.11 Post Digestion Spike Sample Recovery Summary (Form 5B or equivalent) for ICP. This summary shall include instrument printouts for the spiked samples. The summary shall contain the following information: sample matrix, sample result (SR), spiked sample result (SSR), spike added (SA), % recovery (%R), control limits for %R for each analyte, initials of the analyst, data qualifiers and the date of analysis. This is a sample-specific QC element.

4.3.12 Duplicates Summary (Form 6 or equivalent) for ICP and AA. This summary shall include instrument printouts for both the sample and its laboratory duplicate. The summary shall contain the following information: sample matrix, sample result (S), duplicate sample result (D), %solids for both sample and duplicate (if applicable), RPDs and control limits for each analyte, initials of the analyst, data qualifiers and the date of analysis. This is a sample-specific QC element for AML data storage purposes although it may be interpreted as a batch-specific QC for data validation purposes.

4.3.13 Laboratory Control Sample (LCS) Summary (Form 7 or equivalent) for ICP and AA. This summary shall include instrument printouts for the solid LCS sample or spiked blank samples. The summary shall contain

the following information: sample matrix, sample results (S), true values (T), % recovery (%R) and control limits for each analyte, initials of the analyst and the date of analysis. The instrument printout is an acceptable summary for projects that do not require a separate summary form as long as the printout contains all the required information. If LCS is analyzed in duplicate, include %R for both analyses and the RPD for the duplicate analysis. This is a batch-specific QC element.

4.3.14 Method of Standard Addition (MSA) Results Summary (Form 8 or equivalent) for AA. This summary shall include instrument printouts for both the sample and three standard additions. The summary shall contain the following information: sample matrix, sample result without standard addition, sample result for each successive addition, calculated final concentration, initials of the analyst, data qualifiers and the date of analysis. The instrument printout is an acceptable summary for projects that do not require a separate summary form as long as the printout contains all the required information. This is a sample-specific QC element.

4.3.15 ICP Serial Dilutions Results Summary (Form 9 or equivalent). This summary shall include instrument printouts for both the sample and the serial dilution(s). The summary shall contain the following information: sample matrix, initial sample result without dilution, serial dilution sample result corrected for the dilution, %D, initials of the analyst, data qualifiers and the date of analysis. The instrument printout is an acceptable summary for projects that do not require a separate summary form as long as the printout contains all the required information. This is a sample-specific QC element.

4.4 The Wet Chemistry section will review, verify accuracy and compliance and temporarily maintain the following records in a raw data folder for each project and provide the same to the Project and Data Management section after project completion:

4.4.1 For each test performed copies of the sample preparation (e.g. distillation, digestion, etc.) logs and/or instrument analytical logs containing information associated with a specific AML project. Review to determine compliance with technical holding times. Prepare a note describing deviations and recommendations for flagging as appropriate.

4.4.2 Analysis Data Sheet for one or more samples included in the project batch. This will include at a minimum the date of sample digestion, date of sample analysis, analyst initials, sample matrix and the laboratory sample number. The data sheet shall include the test parameter, calibration information, blank information, LCS information, matrix spike information, MDLs and/or reporting levels, analyte amount for each sample, units and data qualifiers. Instrument printouts shall accompany analytical data sheets, if they are available.

4.5 The Sample and Waste Management section will inspect each shipment of samples received by the laboratory, verify accuracy of accompanying paperwork and temporarily maintain the following records in a raw data folder for each project and provide the same to the Project and Data Management section immediately after sample login:

4.5.1 Cooler Receipt Form. For most commercial projects, information contained in the chain-of custody forms is sufficient documentation. Government projects (USACE, EPA, etc.) will require a separate Cooler Receipt Form.

4.5.2 Chain-of-Custody Form properly completed by the laboratory.

4.5.3 Sample Disposal Form for each project.

4.6 The Project and Data Management section will generate and maintain the following records in a filing system consisting of file cabinets, archive boxes, file folders, etc. as applicable:

4.6.1 A draft report based on project data and information provided as described in sections 4.1 to 4.5. The draft report will be reviewed by the Operations Management section and/or the Quality Management section prior to preparing a final report. The draft report or remaining parts of the draft report shall be destroyed after a final report has been generated. Checklists used by reviewers, comments, raw data, etc. shall be kept in the files for a period of up to five years after a final report has been generated.

4.6.2 See section 3.7 for management of final reports and associated raw data.

4.7 The Operations Management section will review, verify and temporarily maintain the following records in a raw data folder for each project and provide the same to the Project and Data Management section before, during and after project completion:

4.7.1 Cooler Receipt and Chain-of Custody Forms.

4.7.2 Draft report with comments directly written on to them.

4.7.3 Approved Final report with initials and/or signature.

4.7.4 Sample disposal form. For each sample that is part of an AML project or SDG, the determination of how that sample will be disposed.

4.7.5 QC Data Verification Form. For each AML project that requires a report, include a completely filled out checklist used to check sample and QC/QA data for adherence to method and project requirements.

4.8 Quality Management section will review, verify and maintain the following records in bound notebooks and/or binders:

4.8.1 QA Data Verification Form. For each AML project that requires additional QA review, include a checklist to check sample and QC/QA data for adherence to method and project requirements.

5.0 QUALITY CONTROL

5.1 The Operations Management section will implement procedures contained in this SOP including any future revisions and management directives that are related to this SOP.

5.2 The Quality Management section will audit data and information storage and retrieval procedures as often as necessary to verify compliance and to receive feedback for its improvement.

STANDARD OPERATING PROCEDURES

No. Q-DataVerification-RC

DATA REDUCTION, REVIEW, VERIFICATION, AND REPORTING PROCEDURES

October 31, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) is intended to provide guidelines for reduction, review, verification, and reporting of laboratory data generated by Analytical Management Laboratories (AML). Project-specific requirements will supersede guidelines included in this SOP. This SOP shall be used in conjunction with management directives, if any, to address changes that may be necessary for rapid compliance with project requirements. Changes that result from technological advances will also be incorporated through management directives.

Achieving the objectives of AML requires the implementation of a system for data reduction, data review, data verification, data reporting and for monitoring the accuracy, precision, and completeness, in analysis. Compliance with data quality objectives stated by the client is assessed from laboratory data on calibration, blanks, duplicates, spikes, spike duplicates, internal standards, and surrogates.

2.0 SUMMARY OF THE PROCEDURE

AML collects voluminous data related to instrument tuning, method-specific initial calibration, calibration verification, analytical results for each environmental sample in an AML work order and associated quality control samples. Copies of relevant data for review, verification and approval of sample data for each method such as batch-specific QC/QA summaries, instrument printouts for samples, etc. are collected into each AML work order data folders. The analysts and the reviewers document their review on checklists designed to determine data limitations as well as the need for re-analysis. The final report and a copy of the checklist used in the final review are kept in the work order data folders.

3.0 DATA REDUCTION

Data obtained by the following method/instrument are directly reportable: Volatile GC/MS, volatile GC, semivolatile GC/MS, ICP metals, AA metals, pH, turbidity, automated colorimetry, etc. Factors that affect the final results such as sample weight/volume, final extract or digestate volume, percent solids and dilution factor are input into the instrument computer and correct results are calculated automatically. Manual calculations may be made if any of these correction factors were unavailable at the time of instrumental analysis.

Data requiring reduction prior to reporting include semivolatile GC, PCBs, BOD, COD, conductivity, manual UV/VIS/IR, residue, titrimetric methods, etc. Spreadsheets are used to input relevant information such as calibration curves, sample weight or volume, dilution factors, final volume of the extracts, percent moisture etc. and correct results are calculated in batches. The following equations are used to calculate results:

$$RF = \frac{A_s}{C_s} \qquad \text{(Equation 1)}$$

where

RF = Response Factor
A_s = Peak area of the standard
C_s = Concentration (µg/ml) of the standard solution

Response Factors (RF) are used in volatile and semivolatile GC analysis of target compounds by external standard calibration methods. Sample concentrations are determined using the following equations for water and soil samples with the assumption that injection volumes are identical for sample extracts and calibration solutions:

$$\text{Amount } (\mu g/L) = \frac{A * V_e * DF}{RF * V_s} \qquad \text{(Equation 2)}$$

where

A = Peak area for the sample extract
V_e = Final volume of the extract (ml) before dilution
V_s = Volume of water sample (L) extracted
DF = Dilution Factor, if any

$$\text{Amount } (\mu g/Kg) = \frac{A * V_e * DF * 100}{RF * W_s * PS} \qquad \text{(Equation 3)}$$

where

W_s = Weight of sample (Kg) extracted
PS = Percent solids expressed in percent

$$RRF = \frac{A_s * C_{is}}{C_s * A_{is}} \qquad \text{(Equation 4)}$$

where

RRF = Relative Response Factor
A_s = Peak area of the standard
C_s = Concentration (µg/ml) of the standard solution
A_{is} = Peak area of the internal standard

C_{is} = Concentration (µg/ml) of the internal standard solution

Relative Response Factors (RF) are used in volatile and semivolatile GC/MS analysis of target compounds by internal standard calibration methods. Occasionally, they are also used in GC analysis. Sample concentrations are determined using the following equations for water and soil samples:

$$\text{Amount } (\mu g/L) = \frac{A * C_{is} * V_e}{A_{is} * RRF * V_s} \qquad \text{(Equation 5)}$$

where

A = Peak area for the analyte in the sample extract
A_{is} = Peak area of the internal standard in the sample extract
C_{is} = Concentration (µg/ml) of the internal standard in the sample extract
V_e = Final volume of the extract (ml) including any dilution
V_s = Volume of water sample (L) extracted

Equation 6 is used to calculate sample amounts in soil and related matrices.

$$\text{Amount } (\mu g/Kg) = \frac{A * C_{is} * V_e * 100}{A_{is} * RRF * W_s * PS} \qquad \text{(Equation 6)}$$

Mean response factors (RF_m) or mean relative response factors (RRF_m) are calculated from multiple determinations of these quantities as follows:

$$X_m = \sum_{i=1}^{n} X_i \qquad \text{(Equation 7)}$$

where

X= is the mean value of n determinations for parameters such as RRF_m and RF_m
Xi = is the individual values for each of n determinations.

Variance (S^2) and standard deviation (S) are calculated using the following equations:

n

$$S^2 = \{1/(n-1)\} \sum_{i=1} (X_m - X_i)^2 \qquad \text{(Equation 8)}$$

$$S = \sqrt{S^2} \qquad \text{(Equation 9)}$$

The coefficient of variation (CV) or percent relative standard deviation (RSD) is calculated as follows:

$$CV = RSD = (100 * X_m)/S \qquad \text{(Equation 10)}$$

The percent difference (%D) between a mean value (X_m) and a single value (X_i) is calculated as follows:

$$\%D = 100 * \{(X_m - X_i)/ X_m\} \qquad \text{(Equation 11)}$$

This equation is used often to verify initial calibrations.

Calibration curves for common wet chemistry parameters are constructed through linear or non-linear regression methods. The most common procedure used is a straight-line (linear regression) plot, which uses the following equation:

$$Y = m * X + C \qquad \text{(Equation 12)}$$

where

Y = measured value such as absorbance
X = concentration of the standard solution (mg/L)
m = slope of the straight line plot
C = Y intercept of the straight line plot

Spreadsheets are used to determine slopes, intercepts and correlation coefficients. Sample concentrations are calculated simply by plugging in the measured value such as absorbance for the sample in equation 12. Equations used for calculating general chemistry parameters vary by the test. These are calculated as described in appropriated methods.

4.0 DATA EVALUATION

4.1 Evaluation Items

When the analysis of a sample set is completed, the results will be reviewed and evaluated to assess the validity of the data set. Review is based on the following criteria.

A. Method Blank Evaluation - The method blank and preparation blank results are evaluated for high readings characteristic of background contamination. If high blank values are observed, the analysis should be halted and laboratory glassware and reagents should be checked for contamination. A high background is defined as a background value sufficient to result in a difference in the sample value greater than or equal to the smallest significant figure known to be true. A reagent blank should contain no greater than three times the method detection limit. See applicable test-specific SOPs for details.

B. Field Blank Evaluation - Field blank results are evaluated for high reading. similar to the reagent and/or method blanks. Field blanks will include equipment rinsates, blank samples exposed to field conditions and trip blanks. If high field blank readings are encountered, the procedure for sample collection, shipment, and laboratory analysis should be checked. If both the method blanks and the field blanks exhibit significant background contamination, the source of contamination is probably within the laboratory. For volatile organic analyses, ambient air in the laboratory and reagents should be checked for possible sources of contamination. High field blank readings for other parameters may be due to contaminated sample bottles or cross-contamination due to sample leakage and poorly sealed sample containers. The laboratory will not evaluate the results for blind field blank samples.

C. calibration standard Evaluation - The daily calibration curve is evaluated to determine linearity through its full range, and that sample values are within the range defined by the low and high standards. If a curve is not linear, sample values must be corrected for nonlinearity by deriving sample concentrations from a graph or by using an appropriate algorithm to fit a nonlinear curve to the standards. See applicable test-specific SOPs for details.

D. Laboratory Control Sample Evaluation - The results of laboratory control sample (LCS), LCS duplicate (LCSD) or quality control check standard (QC) analyses are compared with the true values and the percent recovery of the check standard is calculated. If the recoveries are outside the control limits, the LCS or QC check standard should be reanalyzed to demonstrate that the corrective action has been successful. If reanalysis of the LCS is not successful, the QA Manager should be notified, and reanalysis of all associated samples for the affected parameters may be needed. See applicable test-specific SOPs for details.

E. Surrogate Recovery Evaluation - The results of surrogate standard determinations are compared with the true values spiked into the sample matrix prior to extraction and analysis. The percent recoveries of the surrogate standards must be determined and compared with the control limits established by USEPA and/or by the laboratory. Recoveries must be within control limits for data to be acceptable for volatile and semivolatile organic analyses. See applicable test-specific SOPs for details. Recoveries consistently outside for replicate analyses of the same sample are indicative of matrix related problems.

F. Matrix Spike Evaluation - The observed recovery of the spike versus the theoretical spike recovery is used to calculate accuracy, defined as the percent recovery. The percent recovery value may be plotted on a control chart for the parameter determined. When the percent recovery value exceeds the warning limit for the parameter in question, the QA Manager is notified. If the accuracy value exceeds the control limit, it may be indicative of either laboratory problems or matrix related problem. If the recoveries for LCS or LCSD analyzed with the sample batch are acceptable, matrix related problems are strongly suspected. The sample set may be reanalyzed the same way as described for the laboratory control sample analysis if matrix problems are ruled out. See applicable test-specific SOPs for details.

G. Replicate Standard Evaluation - Duplicate sample analysis for the sample set is used to determine the precision of the analytical method for the sample matrix. The duplicate results are used to calculate the precision as defined by the relative percent difference (RPD). The RPDs should be plotted on control charts. If the RPD exceeds the warning limit, the QA Manager is notified. If the RPD value exceeds the control limit, the sample set may be reanalyzed for the parameter in question if the matrix is determined to be homogenous. If other available QC parameters (surrogates, percent recoveries, RPDs for LCS duplicates, etc.) are in control, matrix heterogeneity is suspected and the data are reported with qualifiers. Allowable precision limits will be specified by the QA Manager and updated periodically following data review.

H. Matrix Spike Duplicate Evaluation – These results, when available, are treated in the same manner as a duplicate result for the purposes of evaluating the precision of the analytical method for the group of sample being analyzed.

I. LCS Duplicate Evaluation – These results, when available are treated in the same manner as a duplicate result for the purposes of evaluating the precision of the analytical method for the group of sample being analyzed.

J. Standard Reference Material Evaluation - Standard Reference Material (SRM) analyses are compared with the true values and acceptable ranges. SRMs are analyzed less frequently compared to LCS. Values outside the acceptable ranges require corrective action to determine the source of error. All sample analyses should be halted pending this evaluation. Following correction of the problem, the SRM should be reanalyzed.

Additional data quality control is accomplished by participation in performance evaluation studies suggested by the US EPA such as the Water Pollution (WP) and Water Supply (WS) programs and programs operated by State or private agencies such as Analytical Products Group (APG) or Environmental Resource Associates (ERA). Successful analysis of these types of performance evaluation samples can be used to attain laboratory certification in both organic and inorganic analyses.

5.0 VERIFICATION AND REVIEW PROCESS

Data verification begins with the processing of data. Data processing can be performed by the analyst who obtained the data or by another analyst. Verification continues with checking that the data processing has been done correctly. The second step in the verification process can be performed either by a second analyst or by the Team Leader. The review process shall be documented by signature (or initials) and date on the documents verified and reviewed.

In general, data will be processed by analyst in one of the following ways: manual computation of results directly on the data sheet or on calculation pages attached to the data sheets; input of raw data for computer processing; and direct acquisition and processing of raw data by a computer. If data are manually processed by an analyst, all steps in the computation shall be provided including equations used and the source of input parameters such as response factors, dilution factors, and calibration constants. The analyst shall sign and date in ink each page of calculations. For data that are input by an analyst and processed by a computer, a copy of the input shall be kept and uniquely identified with the sample numbers and other information as needed. The samples analyzed shall be evident and the input signed and dated by the analyst. If data are directly acquired from instrumentation and processed, the analyst shall verify that the following are correct: sample numbers, calibration constants and response factors, output parameters such as units, and numerical values used for detection limits. The analyst shall sign and date the resulting output. A checklist must be completed for each batch of client samples processed by the analyst.

Data reports must be reviewed to verify that information reported by AML corresponds with processed analytical results. This is first done by the Team Leader and then by the Quality Assurance Manager, Project Manager, or the Lab Director prior to being released by the laboratory. Those who review data reports shall indicate such by signature on the checklists.

5.1 Level I Review

The analyst will draw on his/her experience in the interpretation and use of the internal or routine quality control samples in order to gauge the quality of the analytical results for each batch of samples. The analyst's primary responsibility is for the calibration of the instrumentation (ICAL, CCVs, CCBs, etc.) and incorporation and analysis of the required quality control samples (MB, LCS, LCSD, MS, MSD, etc.). The analyst will verify the sensitivity or responses (SPCC, CCC, RF, CF, etc.) of each instrument, stability of the system, elimination of baseline interferences, etc. Additionally, the analyst will check for the absence of unusually high background due to any reagents (MB, reagent blank, ICB, CCB, etc.) used throughout the analytical process. Once the analyst has verified that the instrument has been calibrated and is responding in the appropriate manner, he/she will proceed to complete the analysis of a batch of samples.

The analyst responsibilities also include the exercising of his/her own expertise in evaluating each batch of samples and the data generated, as well as comparing data for accuracy and precision using the default laboratory quality control limit tables. The analyst will check for bias or accuracy by verifying that the spike recoveries (LCS, LCSD, MS, MSD, PDS, etc.) are within the default limits established by the laboratory for the specific project. The analyst will check for precision by verifying that the replication (LCS duplicate, MS duplicate, sample duplicate,

etc.) of samples (e.g. RPD) is within the prescribed limits established by the laboratory. Any variance greater than the control limit(s) may require rejection, reanalysis, re-extraction and reanalysis or qualification of all analytical results in that specific sample batch.

The analyst will complete a checklist appropriate to the test being performed. A number of checklists are available. At a minimum, the checklist used to present raw data (Table 1) for Level IV reports should be employed. More detailed checklists are included in AML SOPs, which are specific to the test performed. If specific checklists are unavailable for a test, an example Level I checklist shown in Table 2 may be used for each batch of client data submitted by the analyst to his/her supervisor along with a brief statement on the acceptability of the data. Other means of documenting review may also be acceptable. The analyst may have no direct responsibility for rejection of any set of data, unless authorized otherwise by the respective supervisor.

5.2 Level II Review

The Level II review may be provided by a second analyst, a Team Leader or a supervisor who may be responsible for reviewing data generated by analysts within their section. The Level II review will be technically and statistically more rigorous in nature. With regard to surrogate recoveries, the Team Leader will compare recoveries for other tests run on the same sample to determine matrix related problems. The Team Leader will check calculations at the frequency listed in the Level II checklist (Table 3) and document any anomalies either on the test-specific checklists used by the analyst, Level II checklist shown in this document, or directly on the raw data submitted by the analyst. Other means of documenting review may also be acceptable. The Team Leader will order reanalysis as needed but, he will have no direct responsibility for rejection of any set of data that meet most but not all acceptance criteria.

5.3 Level III Review

Level III review will be performed by the Operations Manager, Project Manager, Quality Assurance Director or the Laboratory Director, who will have the authority and/or responsibility to approve, reject or qualify data reported to clients. Although the same accuracy charts used by the Analyst or Team Leader will be used for data assessment, the controlling factor for the acceptability or rejection of data will take into account the overall needs of the project, which may include project-specific data quality objectives, client preferences, turnaround times, etc.. The Level III review will also include a check for project completeness.

Following the Level III review, the Level III reviewer or his designee may provide specific analysts with advance notice of the potential of any system being declared out of control. An attempt will be made to determine the cause of the deviation of the analytical process. When the analyses are complete, the information recorded in the bench books is reviewed by the Analyst and the Team Leader before it is logged onto the computer for report generation. The final report is then printed and used as part of the Level III review. The revised report, following this Level III review is then printed, signed by the Level III reviewer or his/her designee before they are authorized for release to the client. Level III review comments are documented directly on test-specific checklists, custom checklists generated for each AML work order or a generic

checklist similar to that shown in Table 4. Other means of documenting review may also be acceptable.

6.0 DATA RECORDING

Raw data is recorded in bound books and/or by instrument printout. This includes such information as standard curves, matrix spikes, duplicates, reagent blanks, calculations and any notations concerning a given analysis. Preventive maintenance information is kept separate from the analyst's logbook and with the assigned instrument. Raw data associated with each client report will be archived in a separate folder and kept on facility for a minimum of three years after completion of the project. See applicable AML SOP regarding data storage and retrieval procedures.

7.0 DATA REPORTING

The appearance of the report will be consistent with client requirements. At a minimum, the AML report will contain the following information on Level II reports:
Cover Letter
Sample identification number assigned by the sample collector;
Laboratory sample identifier
Date of sample collection
Date (and time, if required) extraction and/or analysis was performed
Initials of the analyst (if required)
Analytical methods employed
Quality Control (QC) batch number (when required)
Results of analyses grouped by sample and reported by parameter
Units as set forth in the referenced method or in client specifications
Reporting limits (RL)
Method detection limits (MDL) and method quantitation limits (MQL), when required
Copy of the original chain of custody form

For Level III reporting, a quality assurance summary report will also be provided which will include the following for each parameter and each QC batch:
Case narrative
Results of sample analyses grouped by parameter and matrix
Quality control data grouped by parameter and matrix
- Method blank results
- Surrogate recovery results and summary with flags for QC outliers
- Laboratory control sample (LCS) and LCS duplicate (LCSD) results (if performed)
- Matrix spike (MS) and matrix spike duplicate (MSD) results (when available)
- LCS and LCSD (if performed) recoveries, QC or ME limits and flags for outliers
- MS and MSD (when available) recoveries, QC or ME limits and flags for outliers
- Summary of laboratory duplicate results (if performed)
- Summary of serial dilution (SD) test results for metals analysis
- Summary of post digestion spike (PDS) test results for metals analysis
- Summary of %D for SD results (when applicable)

Summary of RPDs for laboratory duplicates (when applicable)
Summary of PDS recoveries, QC limits and flags for outliers

Level IV reports (see EPA Contract Laboratory Program Guidelines for detail) will generally contain associated raw data in addition to the Level III reports. Level IV reports will also contain additional analytical information, which are test-specific as described below:
Instrument tune summary and associated raw data for GC/MS analyses
Internal standard areas and retention time summary
Initial calibration summary and associated raw data for all parameters
Calibration verification summary and associated raw data for all parameters
Sample specific work sheets for GC/HPLC analyses
Documentation of IDL, linear range, etc. for metals analyses
Copies of benchsheets used to document sample preparations
Copies of logbook pages containing analytical information
Copies of discussion items between the laboratory and client personnel
Copies of traffic reports
Other miscellaneous items (project-specific)

See applicable AML SOPs for example reports, keeping in mind that reports may vary from client to client and from project to project.

8.0 REFERENCES

8.1 EM 200-1-1, Validation of Analytical Chemistry Laboratories, US Army Corps of Engineers. July 1, 1994.

8.2 EM 200-1-2, Technical Project Planning (TPP) Process, US Army Corps of Engineers, August 31, 1998.

8.3 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001, Appendix I.

8.4 EM 200-1-6, Chemical Quality Assurance, US Army Corps of Engineers, October 10, 1997.

8.5 EM 200-1-10, Guidance for Evaluating Performance-Based Chemical Data, 28 December 2001.

8.6 ER 1110-1-263, Chemical Data Quality Management for Hazardous, Toxic, Radioactive Waste Remedial Activities, US Army Corps of Engineers, April 30, 1998.

8.7 Department of Defense Quality Systems Manual for Environmental Laboratories, Prepared by DoD Environmental Data Quality Workgroup, Appendix DOD-D, "LCS Control Limits", Final Version 2, June 2002.

8.8 United State Navy, Installation Restoration Laboratory Quality Assurance Guide, Chapter 5, Interim Document, February 1996.

8.9 Air Force Center for Environmental Excellence, Quality Assurance Project Plan, Version 3, March 1998.

8.10 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

Analytical Management Laboratories

Table 1 - GENERIC CHECKLIST

(allowable for completion by the analyst in lieu of other checklists)
(can be modified and customized for specific parameters)

_____ Form-I for each sample

_____ Form-IV - QC Association Form with date and initials

_____ QC Summary (Form-I for MB, LCS/LCSD, MS/MSD)

_____ Form-II for surrogate recoveries (when applicable)

_____ Form-III or equivalent for Spike Recoveries (LCS/LCSD, MS/MSD, PDS)

_____ Raw data for project samples

_____ Raw data for QC samples

_____ Initial Calibration (ICAL) Summary and Raw data
CCVs and CCBs – Summary and Raw Data

_____ Sample Preparation sheet (copy) and LIMS printouts

_____ _____

Date **Analyst Initials or Signature**

Analytical Management Laboratories

Table 2 - GENERIC LEVEL I REVIEW CHECKLIST

(recommended to be completed by the analyst in lieu of other checklists, if unavailable)

Parameter: _____ AML Work Order No:_____

#	ITEM TO BE CHECKED	YES	NO	COMMENTS
1	Sample preparation information correct and complete?			
2	Samples extracted and analyzed within technical holding times?			
3	Instrumental analysis information correct and complete?			
4	Instruments in good condition and in acceptable tune?			
5	Initial calibration acceptable?			
6	Continuing calibrations acceptable?			
7	Correct methods/SOPs followed?			
8	Sample analytical results including units correct and complete for all analyses including reruns?			
9	Blank free of analytes?			
10	LCS and/or LCSD recoveries within QC limits?			
11	Surrogate recoveries for all analyses within QC limits?			
12	MS and MSD recoveries within QC limits?			
13	RPDs for LCS/LCSSD within QC limits?			
14	RPDs for MS/MSD within QC limits?			
15	RPDs for laboratory duplicates within QC limits?			
16	Documentation complete?			
17	Special sample preparation and analysis requirements, if any, met?			

General comments, if any:			
_____	_____	_____	_____
Signature of Analyst	Date	Signature of Supervisor	Date

Analytical Management Laboratories

Table 3 - GENERIC LEVEL II REVIEW CHECKLIST

(recommended to be completed by the Team Leader in lieu of other checklists, if unavailable)

Section: _____ **Project No: _____**

For all tests performed by the section on samples from this SDG:

#	ITEM TO BE CHECKED	YES	NO	COMMENTS
1	Correct methods/SOPs followed?			
2	Calibration data scientifically sound, appropriate to the method and completely documented?			
3	QC (blanks, LCS, LCSD, MS, MSD, surrogate, matrix dup. Results /recoveries /RPDs) results within QC limits or guidelines?			
4	Qualitative identification of analytes correct?			
5	Calculations on 10 to 20% of raw data including calibrations and QC samples free of errors?			
6	Additional data checked including the entire data set, if necessary, if errors were detected?			
7	Quantitative results after these checks correct including units?			
8	Documentation complete and correct, including documentation of anomalies in holding times, sample preparation and analysis?			
9	Data package complete and ready for submittal?			
10	Data qualified, if necessary, and ready for incorporation into the final report and/or LIMS?			

General comments, if any:
_____ _____ _____ Signature of Team Leader Date Signature of Project Manager Date

Analytical Management Laboratories

Table 4 - GENERIC LEVEL III REVIEW CHECKLIST

(recommended to be completed by the QA Officer, Project Manager or Lab Director in lieu of other checklists)

Department: _____ **Project No: _____**

For all tests performed by the laboratory on samples from this SDG:

#	ITEM TO BE CHECKED	YES	NO	COMMENTS
1	A. Deficiencies identified in Level I and II reviews corrected? B. All the required analyses performed?			
2	Test data for samples in this WO consistent, free of anomalies and free of errors including unit errors?			
3	Data in compliance with client contractual requirements including special requirements, if any?			
4	Case narrative, if necessary, prepared and reviewed?			
5	A. Electronic Data Deliverables (EDD) required? B. EDD in the required format?			
6	Data in EDD match hardcopy data?			
7	Sample disposal form completed for this SDG in accordance with laboratory policy and client specifications?			
8	Task order exists for billing the analysis?			
9	Invoices completed for this SDG in accordance with contract requirements, including adjustments for non-compliance?			

General comments, if any:

_____ _____

Signature of person signing-off on report Date

CLERICAL CHECKLIST

Date(s) partial reports were faxed or emailed:	
Date final report was faxed or emailed:	
Date final report was sent and the courier used (US Mail, FedEx, Airborne etc.)	
Date invoice was sent and invoice number:	
Signature of the Administrative Asst.:	Date:

END OF THE DOCUMENT

RESTRICTION OF USE AND DISCLAIMER

This document (SOP and/or its appendix) describes systems and procedures that are dynamic and they are subject to revision by the laboratory without notice. Information such as analyte lists, concentrations, detection limits, quantitation limits, QC limits, etc. that are contained in this document are intended to serve a number of programs, projects, and clients. Information contained in this document may be redundant and it may not be consistent with redundant information contained in this document and in the referenced document. Please contact the laboratory if there are any questions.

Copies of this document (electronic or hardcopy) are provided for a limited intended use by the party or parties that are external to AML. Copying and distribution of this document in any form is prohibited without approval by the laboratory.

STANDARD OPERATING PROCEDURES

No. Q-EDD-RD

ELECTRONIC DATA DELIVERABLES

October 31, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

ELECTRONIC DATA DELIVERABLES

1.0 SCOPE AND APPLICATION

1.1 The purpose of this standard operating procedure (SOP) is to describe procedures that are used to determine that analytical data presented in hardcopy form are identical to those contained in electronic deliverables.

1.2 This SOP is not intended to be comprehensive, since formats for EDDs and hardcopy reports are dependent on client specifications, which may also vary from project to project. Clients may also specify more than one format for hardcopy reports. Type of information (fields, round-off procedures, etc.) contained in these reports may vary due to software limitations, laboratory specifications, etc. which may not be addressed by this SOP. User discretion is required in such cases.

2.0 SUMMARY OF THE PROCEDURE

2.1 Data that are presented in both hardcopy form and in electronic deliverables form should have a common electronic source: spreadsheets, databases or the laboratory information management system (LIMS). Data should be finalized before any reports (hardcopy or electronic) are generated. Once reports are generated, alterations cannot be made to one form of report without making similar alterations to the other form. Interim reports shall not be provided to clients requiring reports in both hardcopy and electronic deliverables.

2.2 The LIMS will be the single source of information for all reports, when the Chemware Horizon LIMS is fully implemented. In the interim, a spreadsheet generated by the Data Manager or the QA Manager shall continue to be the source of information for reports in both forms. Hardcopy client reports are reviewed visually against temporary hardcopy reports generated from client EDDs to assure quality.

3.0 INTERIM PROCEDURE

3.1 Generate spreadsheets containing all the fields represented in the hardcopy reports. This includes laboratory sample numbers, field sample numbers and sampling date. Use only information contained in these fields to generate input for EDDs. Repeat manual-entry of information contained in the spreadsheets into the EDDs or additional manipulation of such information after imports from the original spreadsheets that may alter them are prohibited. No alterations can be made to the original spreadsheets after data are exported to the EDDs and hardcopy reports are generated. Generate EDDs and hardcopy reports in accordance with specifications.

3.2 Label spreadsheets with version numbers. Version numbers on the hardcopy and EDDs shall match.

3.3 Print Form I for each sample and for each parameter from the EDDs. Verify all (100%) positive sample results shown on the EDD. Verify dilution factors, MDLs and PQLs for all negative results as a group for each sample.

3.4 Print Form II and VIII for each sample. Verify that control limits are identical for both hardcopy and EDD. Verify all outliers in the hardcopy against output from the EDDs.

3.5 Print Form IIIA (MS/MSD), Form IIIB (LCS/LCSD) for each QC batch. Verify that control limits are identical for both hardcopy and EDD. Verify all outliers in the hardcopy against output from the EDDs.

3.6 Calibration information is currently not included in the EDDs. Information contained in the hardcopy reports should be made with the most up-to-date information found in the laboratory data package.

3.7 Hardcopy sample information (COCs, Cooler Receipt Form, Project Sample Log, etc.) shall be checked against information entered into the spreadsheet.

4.0 LIMS BASED PROCEDURE

4.1 This procedure is currently unavailable. The vendor (Chemware) is currently working on software to generate hardcopy reports and EDDs.

5.0 QUALTIY CONTROL

5.1 Data Manager and/or his/her designee are responsible for controlling access to data in original spreadsheets/LIMS databases. Read only files may need to be created after data entry operations have been completed.

5.2 Operations Manager, QA Director and/or their designee are responsible for verifying data in the hardcopy against data in the EDDs.

6.0 CORRECTIVE ACTION

6.1 Data Manager is responsible for making alterations to the original spreadsheet/LIMS database, when errors and discrepancies are found. This includes the following:
Determination of the source of discrepancies
Creation of later versions of the spreadsheets and/or databases
Controlling access to all versions of data created as a result of the corrective action

6.2 QA Director is responsible for reviewing changes made as a result of the corrective action and in approving such changes.

STANDARD OPERATING PROCEDURES

No. Q-Ethics-RD

Ethics Statement and Agreement

March 6, 2006

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Ethics Statement and Agreement

1.0 SCOPE AND APPLICATION

This Analytical Management Laboratories (AML) standard operating procedure (SOP) is designed to provide management policy regarding the environmental analytical data generation and reporting. AML management and technical staff should adhere to the procedures set forth in this document on ethical conduct.

2.0 SUMMARY OF PROCEDURE

The management staff and employees complete are required to complete and sign a simple ethics statement and agreement form.

3.0 INTERFERENCES

Not Applicable.

4.0 APPARATUS AND MATERIALS

Not Applicable

5.0 REAGENTS

Not Applicable.

6.0 STANDARDS

Not Applicable.

7.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Not applicable.

8.0 HEALTH AND SAFETY

Not Applicable.

9.0 WASTE DISPOSAL

Not Applicable.

10.0 PROCEDURE

10.1 **Laboratory Policy** – The stated policy of Analytical Management Laboratories, Inc. (AML) is to provide quality data to its clients. AML will endeavor to ensure that analytical data generation activities performed by the laboratory will result in the production of data that are defensible, scientifically valid, of known quality, and can be used with a high degree of confidence by the user. AML intends to follow procedures referenced in the following documents: Laboratory Quality Assurance plan (QAP), standard operating procedures (SOPs) that have been developed to supplement the QAP, project specific documents such as quality assurance project plans (QAPP) developed to meet specific project requirements, and client contractual requirements. AML intends to conform to client specifications and regulatory agency guidelines for each project. The Quality Assurance (QA) program is intended to facilitate a sense of importance for quality control and quality assurance practices by managers, supervisors, analysts, and other participants in the data generation process.

10.2 Data integrity and ethics in the generation of data are integral parts of quality data generation program. Deep awareness of the ethical issues by AML employees and the management is essential for data to be used with a high degree of confidence by the user.

10.3 AML employees and management should be committed to the following:

- 10.3.1 Properly collect, preserve and store environmental samples.
- 10.3.2 Maintain adequate chain of custody procedures from sample collection through reporting, data archiving and sample disposal.
- 10.3.3 Use properly trained analysts to analyze samples within holding times using good laboratory and management procedures.
- 10.3.4 Use approved methods, calibration procedures, precision and accuracy control limits to generate data of known quality.
- 10.3.5 Document activities for verification, review, assessment, validation, and reporting of data.

10.4 **Ethics Agreement Form** - Complete the ethics agreement document (Figure 1) as part of the initial orientation and training (Figure 2). Document annual refresher training in attendance logs (Figure 3).

10.5 **Instrument Electronic Audit Functions** – These tools should be used to verify compliance with other SOPs such as those dealing with manual integration, calibration procedures, etc.

11.0 QUALITY CONTROL

Check personnel file for completed ethics agreement form. Have the employees complete a new form, if the file is not complete.

12.0 DATA VERIFICATION AND VALIDATION

Not Applicable.

13.0 REFERENCES

13.1 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 2001.

13.2 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

13.3 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 3, January 2006.

13.4 EM 200-1-2, Technical Project Planning (TPP) Process, US Army Corps of Engineers, August 31, 1998.

13.5 EM 200-1-6, Chemical Quality Assurance, US Army Corps of Engineers, October 10, 1997.

13.6 ER 1110-1-263, Chemical Data Quality Management for Hazardous, Toxic, Radioactive Waste Remedial Activities, US Army Corps of Engineers, April 30, 1998.

13.7 AML Standard Operating Procedures, Q-Training-RE, Technical training.

13.8 AML Standard Operating Procedures, Q-Audit-RD, Laboratory certification and internal quality assurance audits.

13.9 AML Standard Operating Procedures, Q-DataMgmt-RD, Data and information storage and retrieval procedures.

13.10 AML Standard Operating Procedures, Q-DataVerification-RB, Data reduction, review, verification, and reporting procedures.

Analytical Management Laboratories
Ethics and Data Integrity Agreement

Employee Name:	_____
Laboratory Section:	_____
Start Date:	_____
Supervisor:	_____

1. I, _____*(Name of the Employee)*, state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Analytical Management Laboratories.

2. I agree that in the performance of my duties at Analytical Management Laboratories;

    1. I shall not intentionally report data values that not the actual values obtained;

    2. I shall not intentionally report dates and times of data analyses that are not the actual dates and time of data analyses; and

    3. I shall not intentionally represent another individual's work as my own.

3. I agree to inform Analytical Management Laboratories of any accidental reporting of non-authentic data by myself in a timely manner.

4. I agree to inform Analytical Management Laboratories of any accidental or intentional reporting of non-authentic data by other employees.

_____
(Signature)

_____
(Date)

Figure 1. Employee Ethics and Data Integrity Agreement

Analytical Management Laboratories
Employee Orientation and Training Summary

Employee Name: _____

Laboratory Section: _____

Start Date: _____

Supervisor: _____

New Employee Orientation

Training Topic	Date	Trainer
AML General		
AML Employee Benefits (IRS/INS Forms)		
AML Ethics Policy		
AML Customer Service Philosophy		
AML QC/QA Orientation		
AML Health & Safety Orientation		
AML LIMS Orientation		
AML Computer Orientation		

Internal Technical Training

Training Topic or SOP Number	Period	Trainer

External Technical Training

Training Topic	Date	Related Information (describe)

Figure 2. Employee Training Summary

Analytical Management Laboratories

Annual Ethics and Data Integrity Training Log

Date: _____

Provided by: _____

ATTENDEE NAME	ATTENDEE SIGNATURE

Figure 3. Annual Ethics and Data Integrity Training Log

STANDARD OPERATING PROCEDURES

No. Q-Gravimetric-RC

GRAVIMETRIC MEASUREMENTS AND CONTROL OF BALANCES AND OTHER WEIGHT MEASUREMENT DEVICES

October 31, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This Analytical Management Laboratories (AML) standard operating procedure (SOP) is intended to provide guidance for the calibration of balances, which will be used to perform gravimetric measurements.

1.2 This SOP is based on EPA guidance provided in numerous analytical methods and in ASTM Methods D5522, E898, E319, and E617.

2.0 SUMMARY OF METHOD

2.1 The measurement of weight is a fundamental operation of quantitative analysis. Several types of balances are currently in use at Analytical Management Laboratories. These include single-pan analytical balances, precision top loading balance, high capacity top loading balances, and portable balances.

2.2 The procedure to assure accurate gravimetric measurements includes the following steps: daily monitoring of the balances and annual calibration of the weights used in daily monitoring.

2.3 Accuracy is defined as the degree of agreement of the measurement with true value of the quantity measured. Non-linearity is a point-by-point measure of accuracy if the zero point and the full-scale calibration point have been set true.

3.0 INTERFERENCES

3.1 There are many sources of error in weighing an object: unequal balance arms in two-pan balances, incorrect weight standards, changes in moisture or carbon dioxide content, electrification of glass vessels, temperature, buoyancy, draft, dirt, etc.

3.2 Balances and weights should be kept clean using brush, solvent wipes, etc. to minimize interference.

4.0 APPARATUS AND MATERIALS

4.1 Class S or equivalent (Type-I, Class-0 and/or Class-1) weight sets
4.2 Sartorius Analytical Balance - VOA Room
4.3 LECO LB-80 Analytical Balance - General Chemistry
4.4 OHAUS Analytical Plus Analytical Balance - General Chemistry
4.5 Acculab L-Series LA-200 Analytical Balance - General Chemistry
4.6 Tolerance Charts

5.0 REAGENTS

5.1 Type I water

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Not applicable

7.0 HEALTH AND SAFETY

Not applicable

8.0 WASTE DISPOSAL

Not applicable

9.0 PROCEDURE

A number of characteristics such as repeatability, reproducibility, precision, off-center errors, accuracy, linearity, hysteresis, temperature effects, resistance to external disturbances, etc. are measured by the manufacturer. These terms are defined in ASTM Method E898. Balances are used to weigh out known amount of samples and reagents. Therefore, one or more weights are used to test accuracy and linearity on a daily basis. The balances are serviced on an annual basis to provide continued acceptable performance.

9.1 **Laboratory Weights:** Weights are divided into two types (I and II) and seven classes (0, 1, 2, 3, 4, 5, 6, and 7). Maximum permissible tolerances and applications for each type/class weights are shown in Appendix 1. Appendix 1 contains a table that has been reproduced from ASTM Method E617. See below for a description of the types and classes, which replaces National Bureau of Standards Circular 547, Section 1.

9.1.1 Type I weights are of one-piece construction intended for the calibration of weight classes 0, 1, 2, and 3.

9.1.2 Type II weights can be of any design (ring, plug, screw, etc.). These are not commonly used in the laboratory.

9.1.3 Use Type-I, Class-0 weights for checking the weight of Type-I, Class-1 weights.

9.1.4 Use Type-I, Class-1 weights to check the calibration of analytical balances. Type-I, Class-1 weights are equivalent to Class S weights.

9.2 **Balance Tolerance:** According to USACE the measured weight for analytical balances should be within ±0.1% of the true weight. The tolerance for top-loading balances should be ±1.0% of the true weight. However, the tolerance cannot be tighter than the specifications for the weight standard. Use USACE criteria or the applicable tolerance for the type and class of weight used to check the balances.

9.3 **Daily Monitoring:** Clean the balance thoroughly everyday. Make sure that the scale and weights are clean prior to daily monitoring. Place the true (TW) or standard weights (100-g, 5-g, and 0.2-g) successively, close the doors and record (Table I) the observed weights (OW) to four decimal places in grams. Record the most stable value or the mean value (e.g. Leco balance) if reading fluctuates. Calculate the error as the difference between the true weight and the observed weight in grams. Compare the error to the established tolerance and determine need for appropriate corrective action. Record the corrective action taken in the logbook that contains Table I.

9.3.1 The first corrective action is simply being to clean the balance, balance pan and the weights using Type I water, if necessary. Repeat the procedure and record the results at the bottom of the log.

9.3.2 If that does not solve the problem, restrict the use of the balance to a narrow range and call the balance service engineer to check and recalibrate the balance if necessary.

9.3.3 Inform the Operations Manager and the QA Manager immediately about the problem. Leave a note on the balance restricting its use (e.g. Leco balance).

9.4 **Annual Monitoring:** Each balance should be inspected at least annually and the calibration adjusted. Annual monitoring may be postponed if service to restore the balance to specifications has been performed at least annually. The weights should also be calibrated by an outside agency. Contact the following party for service:

Van Dyke Scale Company
2825 Raytown Road
Kansas City, MO 64128-1356
Phone: 816-861-7191
Fax: 816-861-0986

10.0 CALCULATIONS

Not Applicable.

11.0 QUALITY CONTROL

Perform annual monitoring of the balances to control quality of gravimetric measurements.

12.0 REFERENCES

12.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 1, Quality Control, September 1994.

12.2 American Society for Testing (ASTM) Method Designation D5522-99a, Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid.

12.3 American Society for Testing (ASTM) Method Designation E898-88, Standard Method of Testing Top-Loading, Direct-Reading Laboratory Scales and Balances, Reapproved 2000.

12.4 American Society for Testing (ASTM) Method Designation E319-85, Standard Practice for the Evaluation of Single-Pan Mechanical Balances, Reapproved 1997.

12.5 American Society for Testing (ASTM) Method Designation E617-97, Standard Specification for Laboratory Weights and Precision Mass Standards.

12.6 AML Standard Operating Procedures, Q-Water-RA.

12.7 EM 200-1-1, Validation of Analytical Chemistry Laboratories, US Army Corps of Engineers. July 1, 1994.

12.8 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, Appendix I, US Army Corps of Engineers, February 1, 2001.

12.9 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Prepared by DoD Environmental Data Quality Workgroup, Final Version 2, June 2002.

Table I

Balance Calibration Log

Date: _____ Weight Set ID: S01 Analyst Initials: _____

Balance: OHAUS Analytical Plus - Serial Number: 1118133828				
True Weight TW (g)	**Observed Weight OW (g)**	**Error (g) |OW-TW|**	**Tolerance Limits (g)**	**Corrective Action**
100.0000			± 0.100	
5.0000			± 0.005	
0.2000			± 0.0002	

Balance: LECO LB-80 - Serial Number: 363 (Use is restricted to ± 0.001-g accuracy)				
True Weight TW (g)	**Observed Weight OW (g)**	**Error (g) |OW-TW|**	**Tolerance Limits (g)**	**Corrective Action**
100.0000			± 0.100	
5.0000			± 0.005	
0.2000			± 0.001	Restricted Use

Balance: Sartorius Type 1601-MP8-1 - Serial Number: 3310099				
True Weight TW (g)	**Observed Weight OW (g)**	**Error (g) |OW-TW|**	**Tolerance Limits (g)**	**Corrective Action**
100.0000			± 0.100	
5.0000			± 0.005	
0.2000			± 0.0002	

Acculab LA-200 - Serial Number: 80305394				
True Weight TW (g)	**Observed Weight OW (g)**	**Error (g) |OW-TW|**	**Tolerance Limits (g)**	**Corrective Action**
100.0000			± 0.100	
5.0000			± 0.005	
0.2000			± 0.0002	

Other Balances (describe):				
True Weight TW (g)	**Observed Weight OW (g)**	**Error (g) |OW-TW|**	**Tolerance Limits (g)**	**Corrective Action**
100.0000			± 0.100	
5.0000			± 0.005	
0.2000			± 0.0002	

NOTES: _____

Table 1
List of Available Appendices
(Not included in this document)

Appendix Number	Appendix Topic
1	Weight Classes, Maximum Tolerances, and Applications

NOTE:
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APPENDIX TO THE STANDARD OPERATING PROCEDURES

No. Q-Manual Integration-Appendix-RD

APPENDIX TO THE SOP ON MANUAL INTEGRATION AML POLICY AND PROCEDURES

March 7, 2006

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

Table
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1	USEPA Region V Central Regional Laboratory, Chicago, IL SOP # HK014 on Chromatographic Peak Integration Procedures
2	Figures from Appendix A of the USEPA Region V CRL SOP # HK014 Proper Integration Procedures
3	Figures from Appendix B of the USEPA Region V CRL SOP # HK014 Improper Integration Procedures
4	Figures from Appendix C of the USEPA Region V CRL SOP # HK014 Situations Requiring Analyst Intervention for Peak Integration

Appendix 1

USEPA Region V Central Regional Laboratory, Chicago, IL SOP # HK014 on Chromatographic Peak Integration Procedures

Appendix 2

Figures from Appendix A of the USEPA Region V CRL SOP # HK014 Proper Integration Procedures

Appendix 3

Figures from Appendix B of the USEPA Region V CRL SOP # HK014 Improper Integration Procedures

Appendix 4

Figures from Appendix C of the USEPA Region V CRL SOP # HK014
Situations Requiring Analyst Intervention for Peak Integration

STANDARD OPERATING PROCEDURES

No. Q-ManualIntegration-RD

MANUAL INTEGRATION
AML POLICY AND PROCEDURES

March 6, 2006

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) is a guidance document to perform peak integrations for chromatographic systems. Peak integrations include autointegration, which is performed by the instrument/data system and manual integration, which is performed at analyst's discretion. This SOP is applicable for instruments that generate chromatographic peaks.

2.0 SUMMARY OF SOP

Most chromatographic data systems such as the HP Chemstations have autointegration subroutines for data analysis. These subroutines try to find the best integration parameters for the chromatogram and integrate it for the analyst. Autointegration may involve one or more steps; setting up values for the integration parameters or initial events using either default or sample-specific values for area reject, threshold, peakwidth and shoulder detection, etc; establishing baselines, start points and end points for each chromatographic peak; and providing a figure and/or a table of retention times, peak identity, peak heights, and peak areas for each chromatographic peak.

The analyst has the discretion to override autointegration results and to perform these functions manually, if in his/her professional judgment the manual integration results are representative of the actual sample. Procedures to perform manual integrations are not described in this SOP since they are described in applicable instrument manuals and method SOPs. The applicability of the manual integration procedure is discussed.

3.0 INTERFERENCES

Chromatographic problems and interferences occur commonly for gas chromatography (GC), high performance liquid chromatography (HPLC), ion chromatography, and associated mass spectrometric (GC/MS and LC/MS) procedures. Chromatographic interferences are unique for each instrument, column, target compound, sample and/or standard. The analyst has the best judgment in detecting and correcting for potential interferences, which may alter the observed values for samples and/or standards, including QC samples.

4.0 APPARATUS AND MATERIALS

Chromatographic Data Analysis Software

5.0 REAGENTS

Not Applicable.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Not Applicable.

7.0 PROCEDURE

7.1 Manual Integration: Manual integration operations in windows based software such as the HP Chemstation are normally performed using the mouse. The peak(s) to be integrated are highlighted and displayed using the right mouse. The starting point and the end points are defined using the right mouse. Since the starting points and end points are specific for each peak in each chromatogram, the professional judgment of the analyst is final for the initial determination of peak heights and peak areas. The supervisor, team leader, operations manager, laboratory director and the QA Officer have responsibility for review and/or modification of the technical operations to improve representativeness.

7.2 Relationship of the Procedure to Certification: There is a perception among laboratory auditors and validators that the laboratories are systematically abusing chromatographic data systems to manipulate calibration and quality control data that have performance requirements. Consequently, some of the AML reports include the following statements in the case narrative to overcome such perception:

> "*Manual integration operations that have potential to improve accuracy of analysis were performed, as necessary (shown with a "M" flag on chromatograms included in level IV packages) based on visual inspection of peak shapes for each target analyte. All such operations were technically defensible and they were not aimed at meeting the minimum technical requirements of the analytical procedure."*
>
> *"AML certifies that this data package is in compliance with the terms and conditions of this purchase order, both technically and for completeness, for other than the conditions detailed above. Release of data contained in this report has been authorized by the quality assurance manager or his designee, as verified by the signature on the cover letter".*

7.3 Guidance for the Analysts – Prohibited Activities

Manual integration and related activities aimed at meeting the minimum technical requirements of the procedure are prohibited at AML. Some of the prohibited activities are shown below:

7.3.1 Peak shaving and/or peak enhancements to increase or decrease peak areas for tune (DFTPP, BFB, etc.), column check (e.g. endrin/DTT breakdown), etc. to bring them within their QC limits.

7.3.2 Peak shaving and/or peak enhancements to increase or decrease response factors or calibration factors for initial/continuing calibration to bring them within their QC limits.

7.3.3 Peak shaving and/or peak enhancements to increase or decrease surrogate and/or internal standard peak heights or areas to bring them within their QC limits.

7.3.4 Peak shaving and/or peak enhancements to increase or decrease analyte/compound peak areas or heights to bring their concentrations within their recovery limits in LCS/LCSD, MS/MSD, laboratory duplicates and other QC samples.

7.3.5 Other activities that may arouse suspicion from supervisor, QA Director or external

auditors.

7.4 Guidance for the Analysts – Accepted Activities

Manual integration is an acceptable practice that is allowed as long as they are reasonable, technically justified and legally defensible. The analysts are not required to accept all the integration results performed by the instrument. The analyst is neither required nor expected to defend their practice from unreasonable auditors with special requirements of their own as long as they do not engage in prohibited activities. The following are accepted activities and the supervisor should be consulted whenever the analyst is in doubt:

7.4.1 To mitigate an interference detectable from a closely eluting or coeluting analyte or interfering compound for samples and standards when the system is functioning normally.

7.4.2 To address problems arising from minor degradation to the chromatography system directly from the subject sample or from previous samples. Major degradation of the chromatography system should be addressed by other more appropriate corrective action procedures (e.g. sample cleanup, sample dilution, column clipping, cleaning the injector, etc.). Manual integration to overcome degradation is not acceptable for ICALs and ICV. They are also unacceptable for matrix-free batch QC samples (MB, LCS, and LCSD) analyzed immediately after the ICAL/ICV or the beginning CCV unless specifically authorized by the supervisor for rapid turnaround projects requiring screening level data. The matrix-free QC samples or standards (beginning CCV or CCB) should be analyzed using a clean system that is free of degradation. Manual integration to overcome degradation is acceptable for middle and ending CCVs, MS and MSD samples as long as the analyst exercises sound judgment and they are scientifically justified. Fear of an auditor should not result in the unnecessary wasting of resources.

7.4.3 To address problems from automated integration from peak tailing, if the chromatographic system was functioning normally at the beginning of analysis.

7.5 Examples

Appendix 1 contains the text of standard operating procedure employed at the USEPA Regional Laboratory, Chicago, IL, which contains a number of figures illustrating peak integration procedures. The figures from the referenced SOP are included in Appendix 2 to 4.

7.5.1 Figure A-1 and A-2 in Appendix 2 describe properly integrated single peak and unresolved peaks respectively. Figure A-3 describes proper integration procedures to remove interfering peaks. Figure A-4 describes the use of peak skimming, which may require more sophisticated software than those available at the laboratory.

7.5.2 Figures B-1 to B-4 in Appendix 3 describe improper peak integration procedures that illustrate peak shaving procedures. Figure B-5 illustrates improper peak enrichment procedure.

7.5.3 Figures C-1 to C-4 in Appendix 4 contain examples of situations where manual integration requiring analyst discretion is recommended.

8.0 CALCULATIONS

Not Applicable.

9.0 QUALITY CONTROL

The supervisor is responsible for performing random audits to detect manual integration practices that may require review and resolution. The supervisor and/or the Team Leader is responsible for bringing such issues to the attention of QA Director, who will have the final authority to approve/disapprove manual integration procedures requiring review.

9.1 Electronic audit functions available in the instrumentation may be used by the supervisor or the auditor to verify compliance.

9.2 Consequences to the analysts for non-compliance with this SOP will be severe and it will include immediate termination from the company.

9.3 While AML will vigorously defend itself and its employees, it is AML policy for the organization and its employees to cooperate fully with regulators, auditors and investigators.

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10.1 EM 200-1-1, Validation of Analytical Chemistry Laboratories, US Army Corps of Engineers. July 1, 1994.

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10.14 Standard Operating Procedure – Chromatographic Peak Integration Procedures, CRL SOP # HK014, USEPA Region V Central Regional Laboratory, Chicago, IL, 11/9/1999.

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STANDARD OPERATING PROCEDURES

No. Q-ManualIntegration-RD

MANUAL INTEGRATION
AML POLICY AND PROCEDURES

March 6, 2006

APPROVED:

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Operations Manager, Analytical Management Laboratories Date

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4	Figures from Appendix C of the USEPA Region V CRL SOP # HK014 Situations Requiring Analyst Intervention for Peak Integration

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices. The appendices may be available in hardcopy form or as portable document electronic files (pdf).

STANDARD OPERATING PROCEDURES

No. Q-MDL-LLR-RL-MQL-RC

INSTRUMENT DETECTION LIMITS (IDL) METHOD DETECTION LIMITS (MDL) LOWEST LEVEL FOR REPORTING (LLR) REPORTING LIMITS (RL) METHOD QUANTITATION LIMITS (MQL) AND INITIAL DEMONSTRATION OF PERFORMANCE (IDP)

October 31, 2005

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INSTRUMENT DETECTION LIMITS (IDL), METHOD DETECTION LIMITS (MDL), LOWEST LEVEL FOR REPORTING (LLR), REPORTING LIMITS (RL), METHOD QUANTITATION LIMITS (MQL) AND INITIAL DEMONSTRATION OF PERFORMANCE (IDP)

1.0 SCOPE AND APPLICATION

1.1 This Analytical Management Laboratories (AML) standard operating procedure (SOP) is used to provide guidance for the determination of the following items: Instrument detection limits (IDL), method detection limits (MDL), lowest level for reporting (LLR), reporting limits (RL), estimated, practical, or method quantitation limits (EQL/PQL/MQL), sample quantitation limits (SQL), and initial demonstration of performance or capability (IDP or IDC) for organic and inorganic analyses.

1.2 This document also provides guidance for demonstrating method performance at AML before it can be used to analyze environmental samples.

1.3 Procedures outlined in this SOP are based on the U.S. Environmental Protection Agency (EPA) methods and U.S. Army Corps of Engineers (USACE) guidance documents. Definitions and procedures have been reproduced verbatim from EPA and USACE documents when necessary to facilitate adherence to acceptable methods.

2.0 DEFINITIONS

2.1 The EPA SW-846 definition of method detection limit (MDL) is shown in Table I. The EPA SW-846 definition of MDL is consistent with the definition contained in 40 CFR Part 136 Appendix B, Federal Register dated October 26, 1984 and subsequent revisions. The USACE definition of MDL (See reference cited in Section 7.3) is very similar.

2.2 MDLs at a minimum are matrix and analyte/compound specific. Since the process for MDL determination may include one or more sample extraction, one or more sample cleanup (more than one option may be applicable) and one or more instrumental analysis (different instruments, different chromatography columns) steps, there is potential for MDL variability even for a single matrix. In order to simplify MDL determination, the procedure included in 40 CFR Part 136 Appendix B is "device- or instrument-independent". A pragmatic approach is used at AML to balance the need for accurate MDLs for each situation against the associated economic consequence and/or the potential for confusion when multiple MDL sets are provided for a single analytical method. MDLs are generally reported to two or three significant figures or decimal places. Default MDLs determined under standard or default conditions (applicable to MB, LCS, LCSD, and other interference-free matrices) are adjusted based on actual sample weight or volume, percent solids (when applicable) and extract dilution employed for instrumental analysis. The adjusted MDLs, which are shown in sample reports, may be defined as sample detection limits (SDL). Adjusted MDLs are reported for USACE projects.

2.3 The term, instrumental detection limit (IDL) is generally used at AML with metals analysis performed using ICP or AA instruments. It is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte or compound concentration in solution is greater than zero and it is determined from instrumental analysis of a solution containing the analyte or compound. IDLs are determined using a combination of statistical and administrative procedures described in EPA Contract Laboratory Program (CLP) documents. These procedures have been simplified at AML so that they are similar to the procedures employed for MDL determination. IDLs are reported to two significant figures or to two decimal places. IDLs are not corrected for dilution.

2.4 The EPA SW-846 defines the estimated quantitation limit (EQL) as five to ten times the MDL as shown in Table I. EPA has also defined minimum level of quantitation (ML) in Chapter 3, Section 3.4.1.1of the December 1996 EPA Guide on Method Flexibility and Approval document (See Section 7.4). The ML is established by multiplying the MDL by 3.18 and rounding to the number nearest to (1, 2, or 5) x 10^n, where n is a positive or negative integer. The purpose of rounding is to allow instrument calibration at a concentration equivalent to the ML without the use of unwieldy numbers. The use of 3.18 results in an overall standard deviation multiplier of 10, which is consistent with the American Chemical Society's (ACS) limit of quantitation (LOQ), which is described in an article by P.S. Porter (Section 7.5).

2.5 USACE defines MQL as "the lowest level that can be reliably measured by routine laboratory operating conditions within specified limits of precision and accuracy". USACE has the following guidance for MQL, which has been reproduced verbatim from the referenced document (Section 7.3): "Due to the significant amount of error (approximately 100%) associated with results calculated at the MDL and the fact the MDL may not be attainable within project matrices, the method quantitation limit (MQL) is established at a factor of five to ten times the MDL for the majority of target analytes, but no lower than three times the MDL for any target analyte. The statistical error (±20-30%) associated with this area of the calibration curve is notably reduced from the MDL. The appropriate factor applied to the MDL to establish the MQL is based upon the acceptable amount of error the data user is willing to accept for the data generated. Ideally this MQL should have an associated error comparable to the method prescribed CCV acceptance limits. This may not be feasible however, due the lower concentration range of interest. This approach, however, may not be appropriate for multi-component target analytes. Due to the identification of multi-component target analytes (e.g., polychlorinated biphenyls (PCBs), chlordane, toxaphene, gasoline, etc.) being based upon a recognizable pattern, the MQL should be based upon the MDL as well as the concentration at which the pattern is reliably "identifiable". Thus the MQL represents the value at which the laboratory has demonstrated the ability to reliably quantitate target analytes within a prescribed performance criterion for the method, and establishes the lowest concentration at which the data may be reported without qualification as an estimated value (i.e., J-flag). In the absence of project-specific requirements to the contrary, USACE requires the following: The MQL is set at the lowest standard

used for the initial calibration curve (or low-level calibration verification standard) or higher for each target analyte. The lowest standard or low-level calibration verification standard must be at least three times the MDL or greater. Target analyte values detected and reported below the MQL must be flagged as an estimated quantity (i.e., J-flag)".

2.6 The term, method quantitation limit (MQL) is employed at AML to describe ML, MQL, LOQ, PQL and EQL. Since the definitions (Sections 2.3 and 2.4) are quite similar, MQL will be set at levels that are equal to or greater than 3.18 times the applicable MDL. PQLs for a majority of the target analytes in multi-analyte methods should be in the 5 to 10 multiples of their respective MDLs, although they could be lower (not recommended) or higher (preferred) for some target analytes. MQLs are generally reported to two significant figures or to two decimal places. Default MQLs determined under standard or default conditions (applicable to MB, LCS, LCSD, and other interference-free matrices) are adjusted based on actual sample weight or volume, percent solids (when applicable) and extract dilution employed for instrumental analysis. The adjusted MQLs, which are shown in sample reports, may be defined as sample quantitation limits (SQL). Adjusted MQLs are reported for USACE projects.

2.7 For all analyses in which a multi-point calibration curve is employed, MQL should be selected as the lowest non-zero standard in the calibration curve. A higher point may be selected for some target analytes when additional calibration points (e.g., six points) than those required (e.g., 5 points) by the method are available.

2.8 For multi target organic analytical methods (e.g. SVOCs), the laboratory may use cut-off levels that may be greater or less than individual target analyte MDLs. A single cut-off level may be used for all target analytes. This cut-off level is a uniform low level for reporting (LLR). When sample concentration for an analyte is greater than LLR, the compound is coded as present only if it is also greater than the associated adjusted MDL. Otherwise, it is coded as not detected (U code). When the sample concentration is less than LLR, it is coded as not detected (U code), even if the observed level is greater than the adjusted MDL. The uniform low level for reporting (LLR) is included in report headers along with information such as dilution factors, sample weight, etc. LLR is sample specific but not target analyte specific. LLRs are normally corrected for percent solids, sample size and dilution. LLRs may be used as optional reporting tools by the laboratory.

2.9 USACE has provided guidance (See Sections 3.3.7.3 and 3.3.7.4 of reference cited in Section 7.3) for method reporting limit (MRL), which is intended for project planners. Some laboratories use the term reporting limit (RL) synonymously with quantitation limits. Due to the confusion associated with this term, reporting limits (RL) or method reporting limits (MRL) are not used by AML for USACE projects.

2.10 The reporting limit (RL) used by AML is unrelated to the USACE MRL. Reporting limits (RL) are used by AML to report data for non-USACE projects. Reports for USACE projects are generated using the laboratory information system (LIMS). Reports for non-USACE (other) projects do not employ the Oracle database of the LIMS. Reporting limits (RL) for the non-USACE projects replace MDLs and MQLs that are employed for USACE and similar projects. Reporting limits are not adjusted

for minor variations from default sample size, percent solids, insignificant sample dilutions, etc. Reporting limits equal unadjusted MQLs in most cases. Target analyte present at levels that are lower than the RL are reported, as "ND" not detected.

2.11 Statistical parameters: The objective of statistical inference is to draw conclusions or make decisions about a population (e.g. total number of measurements possible for a laboratory sample) based on a sample (e.g. number of measurements (n) performed for the same laboratory sample) selected from the population.

2.11.1 For most environmental chemical analysis, sampling without replacement is applicable since sample is often consumed in the analysis.

2.11.2 The sample mean or average for "n" measurements, sample variance (S^2), and sample standard deviation (S) are defined in Table 1.

2.11.3 The degrees of freedom (ν) for "n" measurements in "n-1".

2.11.4 If the probability distribution of the population is known, the sampling distribution, which is the probability distribution of the various statistics computed from sample data, can be determined. Normal distribution is assumed for the population of laboratory analytical data for a sample.

2.11.5 When the number of degrees of freedom approaches infinity, the t-distribution (Student's t value) of sample results reduces to standard normal distribution (See Section 7.6 for more details). A table of percentage points of the t distribution is included in Appendix 1. A condensed table is also included in Table 1.

2.11.6 An interval estimate of a parameter is the confidence interval between two statistics that includes the true value of the parameter with specified confidence or probability. The factor $1-\alpha$ is called the confidence coefficient. For 99% confidence, $\alpha=0.01$ for one-sided interval. For 95% confidence, $\alpha=0.05$ and for 90% confidence, $\alpha=0.1$. One-sided interval with 99% confidence ($\alpha=0.01$, $(1-\alpha)=0.99$ or 99%) is used for defining MDL as shown in Table 1. See Appendix 1 for the distribution of percentage points for various degrees of freedom (ν) and confidence (α) values.

2.11.7 The ratio of the variances (S_1^2 and S_2^2) of two sample normal distributions is used to determine whether two samples 1 and 2 of size n_1 and n_2 and degrees of freedom ν_1 and ν_2 respectively have the same population variance. The F-ratio (S_1^2/ S_2^2) is calculated by substituting the larger variance into the numerator and the other into the denominator. The computed F-ratio is then compared with the F-ratio found in a table (See Appendix 2) for $\alpha=0.1$. For example, the F-ratio for two MDL determinations ($n_1 = n_2 = 7$; and $\nu_1 = \nu_2 = 6$) is 3.05. If the observed F-ratio is less than the ratio in the Table, both samples are considered to be representative of the same population and the variances from the two sample distributions may be pooled as described in the following section.

2.11.8 If F-ratio criteria described in Section 2.10.7 are met, the variance can be pooled to calculate better estimates of the population standard deviation. Calculate S_{pooled} as follows:

$$S^2_{pooled} = \frac{\{[(n_1 - 1) * S_1^2] + [(n_2 - 1) * S_2^2]\}}{(n_1 + n_2 - 2)} \quad (1)$$

$$S_{pooled} = [S^2_{pooled}]^{1/2} \quad (2)$$

2.11.9 When pooled standard deviations are used for MDL determination, t values should reflect the larger degrees of freedom. For example, if $n_1 = n_2 = 7$ and $\nu_1 = \nu_2 = 6$, the t value for 12 degrees of freedom ($\nu = \nu_1 + \nu_2 = 12$) with 99% confidence from Appendix 1 is 2.681. MDL for this case should be calculated as shown below;

$$MDL = 2.681 * S_{pooled} \quad (3)$$

3.0 PROCEDURES

3.1 Instrumental Detection Limits (IDLs)

3.1.1 The IDLs are determined quarterly (every three calendar months). Analysis to determine the IDLs should be normally scheduled during the last week of March, June, September, and December of each year for the IDL data to be used during the following quarter. The following procedures apply only to metals analysis by ICP-AES, ICP-MS, and GFAA.

3.1.2 Using historically determined IDLs for the instruments (TJA ICAP62, PE ELAN-6100 or PE-Zeeman-GFAA) prepare standard solutions containing approximately 1 to 5 times the determined IDL levels or water MDL levels.

3.1.3 Perform seven to nine consecutive instrumental analysis using procedures that are normally used for sample analysis. Calculate mean and standard deviation for seven replicates.

3.1.4 Calculate IDL using procedures that are described for MDL, which are described in Section 3.2.5.

3.1.5 IDLs may be calculated for instrumental analysis other than those mentioned in Section 3.1.1 using a similar procedure.

3.2 Method Detection Limits (MDLs)

The MDLs are normally determined annually. The MDLs may need to be determined more frequently if major changes have been made to sample preparation, instrumental analysis, type of instrument or the instrument model. USACE has the following guidance (reproduced verbatim) for MDL verification (See Section 7.3) an alternative to frequent and expensive MDL determination: "To ensure that reasonable MDL values are determined, the laboratory shall analyze an MDL check sample by spiking an interference free matrix with all target analytes at about two times the determined MDL. The MDL check sample shall be taken through the same process used initially to establish the MDL values. If any of the target analytes are not detected, then the MDL study shall be modified and repeated for the failed

target analytes, until the MDL check sample is detectable. The laboratory may then verify continued method detection capability by analyzing the MDL check sample on a quarterly basis, in lieu of the annual MDL study. When multiple instruments or confirmation columns are used for the same method, separate MDL studies may be replaced by the analysis of an MDL check sample on all instruments/columns. The MDL check sample shall be analyzed after major instrument maintenance, or changes in instrumentation or instrumental conditions to verify the current sensitivity of the method. When low-level detection is critical, it is suggested that the laboratory perform a method detection limit study or an MDL check sample on project-specific samples at project start-ups in order to more accurately assess the sensitivity within that project matrix". The following procedures, which are based on 40CFR Part 136-Appendix B applies to the MDL determination for all analytes and compounds.

3.2.1 Step 1: Make an initial estimate of the method detection limit. Initial estimates may be based on one or more of the following:

3.2.1.1 Historic data. Historic data may include data collected by the laboratory, data available from studies performed by other laboratories and data contained in published methods.

3.2.1.2 The concentration value that corresponds to an instrumental signal to noise (S/N) in the range of 2.5 to 5.

3.2.1.3 The concentration equivalent of three times the standard deviation of replicate measurements (use a minimum of three replicates) of the analyte solution corrected for concentration/dilution factors for sample preparation.

3.2.1.4 The low concentration range where there is a significant change in sensitivity in calibration curves.

3.2.1.5 Instrumental limitations

3.2.2 Step 2: Prepare a blank matrix that is free of target analytes and other interferents. Reagent water may be used as the blank matrix for MDL determination in aqueous samples. Laboratory sand or drying agents such as anhydrous sodium sulfate may be used as blank matrix for the MDL determination of organic compounds in soil, sediment and waste matrices. Teflon boiling chips or clean sample digestion containers may be used as blank matrix for the MDL determination of inorganic analytes in soil, sediment and waste matrices. NIST or other commercially available MDL samples for each matrix may be used for non-aqueous matrices.

3.2.3 Step 3: Prepare seven identical laboratory standards in the blank matrix of interest by spiking each analyte at 1 to 5 times the estimated MDL. Label these standards as MDL-1, MDL-2, etc. Additional replicates may be included as insurance for accidental sample loss in preparation. If historic data from the laboratory is used to estimate MDL, use the most recent MDL data meeting the spike criteria. Inflated MDL may result if sample concentrations are many times the real MDL (e.g. >5 for reagent water and >10 for other matrices).

3.2.4 Step 4: Process the MDL standards through the entire analytical procedure. If multiple instruments are used for an analytical procedure, sample extracts may be analyzed on each instrument. Make all computations according to the defined method with final results in the method reporting units. Blank subtraction, which may be applicable for some analytes is discouraged at AML. However, for some highly sensitive instruments (e.g. ICP-MS), blank contamination may become a problems due to the routine non-availability of water that is purer than Type I. The well-known blank concentration (mean blank concentration over a period of time) should be added to the calculated MDL while assigning actual MDLs for certain analytes (e.g., Ca, Mg, Na, etc.) that cannot be removed completely from calibration blanks.

3.2.5 Step 5: Calculate the variance (S^2), standard deviation (S), and the mean or average x for the seven measurements using equations shown in Table 1. An Excel spreadsheet may be used to perform calculations. The MDL is calculated by multiplying S by 3.14, the student t value for 7 measurements at the 99% confidence level. If more than seven measurements were made, appropriate student t values shown on Table I may be used instead of 3.14. The preferred approach is to throw obvious outliers based on visual inspection (e.g. the high and/or low points when 9 data points are available) of the data or to throw two data sets at random.

3.2.6 Step 6: Second or later iterations. This step is used to fine tune the MDLs by pooling statistical parameters as available MDL data increases with time. However, F-ratio of the variance (S^2) for the current set should be compared to that for the previous set for pooling to be acceptable.

3.2.6.1 Calculate F-ratio as described in Section 2.10.7 and compare the ratios with those in the Table contained in Appendix 2 for acceptability.

3.2.6.2 Calculate pooled standard deviation and the associated degrees of freedom as described in Section 2.10.8.

3.2.6.3 Calculate MDL as described in Section 2.10.9 using t values shown in Appendix 1.

3.3 Method Quantitation Limits (MQLs)

3.3.1 Once MDL is determined, MQL is established by multiplying MDLs by a minimum factor of 3.18 as described in Section 2.6. Higher multipliers greater than 3.18 but less than 10 may be used to for rounding unwieldy concentration values to more uniform numbers for most target analytes. MQLs that are greater than 10 times the statistically determined MDLs are permissible when the standard deviations are unusually low relative to the sensitivity in the professional judgment of the analyst. Once MQL is established, it will be used as the lowest calibration point.

3.3.2 The concentration of the calibration solutions are typically handled in units such as ng/mL, ug/L, mg/L, etc. For most environmental methods, a calibration curve is used to calculate sample concentrations in both water and

soil matrices. The default MQL in sample matrices other than reagent water are calculated from the concentration of the lowest non-zero standard (X_l), the default initial weight (W_i) or volume (V_i) of the sample and the final volume of the sample extract (V_f) for the method. Actual sample weights and other correction factors such as those for percent solids (PS), dilution (D), etc. should be used to calculate adjusted MQLs (sample quantitation limits) using the following equations for water (eqn. 4) and soil (eqn. 5) matrices:

$$MQL = \frac{X_l * V_f * D}{V_i} \quad (4)$$

$$MQL = \frac{X_l * V_f * D * 100}{W_i * PS} \quad (5)$$

Where,
X_l is the concentration (ng/ml or ug/L) of the lowest non-zero calibration standard;
V_f is the final volume of the sample extract (ml) for the method;
Vi is the initial volume of the sample (ml) for the method;
W_i is the initial weight of the sample (g) for the method; and
D is the correction factor for dilution
PS is percent solids for non-aqueous samples

3.3.2 The MQLs (ug/L or ug/kg) for metals analysis by ICP-AES are determined by analyzing low standards (e.g. CRI solutions for ICP-AES), which should contain concentration of the target analytes (e.g. see Table II) at their MQL. The concentration of the elements in the MQL solution for ICP-AES are either equal to or close to their concentrations in the EPA CLP CRI solution, with the exception of arsenic, lead, selenium and thallium, which are present at concentrations that are suitable for their analysis by ICP-AES. The recovery of the MQL solution is used for calibration verification at the MQL. For MQL to be verified, the measured values should be within ±20% of their true values.

3.4 Reporting Limits (RLs)

USACE method reporting limits are described in Sections 2.9. AML reporting limits, which are closely related to AML MQLs are described in Section 2.10. Reporting limits are used to minimize reporting potential false positives associated with reporting estimated sample amounts below MQL. They also provide fast track reporting for projects requiring rapid turnaround analysis. Reporting limits (RL) are not normally used for USACE projects.

3.5 Lowest Level of Reporting (LLR)

The lowest level of reporting (when used) is related to MDLs and MDL check sample analysis, which is described in Section 3.6. For multi target analyte organic analytical

methods such as SVOCs analysis by SW-846 method 8270C, the laboratory may use cut-off levels that may be greater or less than individual target analyte MDLs in instrument quantitation reports. A single cut-off level may be used for all target analytes. This cut-off level is a uniform low level for reporting (LLR).

3.5.1 When sample concentration for an analyte is greater than LLR, the compound is coded as present only if it is also greater than the associated adjusted MDL. Otherwise, it is coded as not detected (U code). When the sample concentration is less than LLR, it is coded as not detected (U code), even if the observed level is greater than the adjusted MDL.

3.5.2 The uniform low level for reporting (LLR) is included in report headers along with information such as dilution factors, sample weight, etc. LLR is sample specific but not target analyte specific. LLRs are normally corrected for percent solids, sample size and dilution. LLRs may be used as optional reporting tools by the laboratory.

3.5.3 As a general rule, LLRs are normally determined by rounding up the highest MDL for the target analytes to a round number. The LLR must be less than PQL. Professional judgment should be used to determine LLRs.

3.5.4 The use of LLR is strictly an optional procedure that may be used in selected projects to simplify laboratory analysis and reporting.

3.6 MDL Check Sample Analysis

Prepare a MDL check sample by spiking an interference free matrix with all target analytes at approximately two times the calculated MDLs. The MDL check sample should be taken through all the preparative and determinative steps used to establish the calculated MDL values, to very a response is detected. If any of the target analytes are not detected in the MDL check sample, then the concentration of the affected compounds should be increased in another MDL check sample and the study repeated until the failed analytes are detectable. The increased concentration for the affected analytes in the second check sample, which gives a positive detection should be defined as the method MDL. Since it may not be practical to prepare a custom MDL check sample at different levels for different types of compounds for multi-analyte methods (e.g. VOCs and SVOCs), the MDL check sample analysis may be performed at two or more levels that encompass the MDLs of the target analytes.

3.6.1 Verify continued method detection limit and reporting limit capability by analyzing the MDL check sample on a quarterly basis, in lieu of the annual MDL study.

3.6.2 When multiple instruments or confirmation columns are used for the same method, separate MDL studies may be replaced by the analysis of a single MDL check sample on all instruments/columns. The MDL check sample should be analyzed after major instrument maintenance, or changes in instrumentation or instrumental conditions to verify the current sensitivity of the method.

3.7 Initial Demonstration of Proficiency

Determination of IDL, MDL, LLR, MQL, etc. are processes that are performed both initially before any client samples are analyzed and at specified intervals for evaluating method performance. The initial precision and recovery test (IPR) is used for initial demonstration of the laboratory's capability to produce results that are at least as precise and accurate as results from practice of the method by other laboratories and/or the reference method. The IPR test consists of analyzing at least four replicate samples of a reference matrix spiked with the analytes of interest and with either surrogate compounds or, for isotope dilution analysis, labeled compounds. Typically, the concentration of the target analytes in the spike solution is approximately five times the MQL. The spiked samples are carried through the entire analytical process and the observed mean recoveries and standard deviation are compared to those described for the reference method. The following procedures should be used at AML for the initial demonstration of proficiency:

3.7.1 Determine MDLs for target analytes as described in Section 3.2.

3.7.2 Establish MQLs as described in Section 3.3.

3.7.3 Establish calibration curve that includes a calibration blank, one standard at the PQL, another standard between two and five times the PQL, a third standard at five to ten times the PQL and additional standards as necessary to meet method requirements.

3.7.4 Prepare a spiking solution that can be used to spike reference matrix with the target analytes at two to five times the MQL.

3.7.5 Prepare and analyze seven replicates (four replicates at a minimum) of the spiked reference matrix using procedures to be followed for actual environmental samples.

3.7.6 Calculate sample concentrations and analyte recoveries for each replicate. Calculate mean recoveries, standard deviation (S) and relative standard deviation (%RSD = mean/S in %)

3.7.7 Compare mean percent recoveries (%R), S, and %RSD with those described in the reference method. If they are within the range for accuracy (default = 70 to 130% for %R) and precision (default = 15% for RSD), the laboratory can analyze client samples. Otherwise, correct the problem and repeat the spiked sample analysis.

3.7.8 Spiked samples prepared as described in this section may also be used as laboratory control samples (LCS) for the demonstration of ongoing precision and recovery.

4.0 CALCULATIONS

4.1 Calculations are described in Section 3, Procedures. Excel spreadsheets are used to perform statistical calculations and to determine MDLs. Employees should contact the Laboratory Manager and/or the QA manager for specific instructions, examples, software diskettes, etc. on using templates that have been set up for each method. Instructions, diskettes, etc. to generate data contained in Appendices 3 and 4 of this SOP are available only for distribution within AML

for internal use. Due to the proprietary nature of the information, copies will not be made available to parties outside the organization.

4.2 Copies of the complete SOPs including all applicable appendices will be made available to on-site auditors for their review and verification.

5.0 SUMMARY OF IDLs, MDLs, LLRs, and MQLs for SPECIFIC METHODS

5.1 Excel spreadsheets should be used to document IDLs, MDLs, RLs and common PQLs for each test in each matrix. Summarize as shown in Appendices 3 and 4. Summaries are included in Appendix 3 of this SOP for aqueous matrices and Appendix 4 for soil, sediment and waste (solid) matrices. Hardcopies and/or electronic versions of data contained in these documents should not be released to outside parties without approval by one of the following: Laboratory Manager, QA Manger, Laboratory Director, or the QA Director.

5.2 Complete copies of the SOPs including all applicable appendices should be made available to on-site auditors for their review and verification.

6.0 IMPLEMENTATION

6.1 Team leaders are responsible for gathering and tabulating information necessary to calculate IDLs, MDLs, LLRs and MQLs for tests in their technical area.

6.2 The QA Manager and the Operations Manager are responsible for performing the necessary calculations and for making policy decisions related to the implementation of these parameters during day to day analysis.

6.3 The QA Manager is responsible for maintaining a current copy of these parameters for use with client project plan preparation and for monitoring the overall performance of the methods at the laboratory.

6.4 The QA Director is responsible for formulating QC/QA policies and AML practices regarding IDL, MDL, LLR, and MQL.

7.0 REFERENCES

7.1 Test Methods for Evaluating Solid Waste, Volume 1: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Section A, Chapter 1, Quality Control.

7.2 Definition and Procedure for the Determination of the Method Detection Limit - Revision 1.11, Appendix B to 40 CFR (7-1-95 Edition) Part 136.

7.3 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, 1 February 2001, Appendix I.

7.4 Guide to Method Flexibility and Approval of EPA Water methods, Prepared by Analytical Methods Staff, Engineering and Analysis Division, Office of Science and Technology, Office of Water, U.S. EPA, Washington, DC, December 1996, Chapter 3.

7.5 P.S. Porter, et.al., Environ. Sci. Technol., 22 (1988).

7.6 Introduction to Statistical Quality Control by Douglas C. Montgomery, John

Wiley, 1985.

7.7 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

TABLE I

ESTIMATED QUANTITATION LIMIT (EQL):	The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL analyte concentration is selected as the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs in SW-846 are provided for guidance and may not always be achievable.
METHOD DETECTION LIMIT (MDL):	The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. For operational purposes, when it is necessary to determine the MDL in the matrix, the MDL should be determined by multiplying the appropriate one-sided 99% t-statistic by the standard deviation obtained from a minimum of three analyses of a matrix spike containing the analyte of interest at a concentration three to five times the estimated MDL, where the t-statistic is obtained from standard references or the table below. No. of samples: t-statistic 3 6.96 4 4.54 5 3.75 6 3.36 7 3.14 8 3.00 9 2.90 10 2.82 11 2.76 Estimate the MDL as follows: Obtain the concentration value that corresponds to: a) an instrument signal/noise ratio within the range of 2.5 to 5.0, or b) the region of the standard curve where there is a significant change in sensitivity (i.e., a break in the slope of the standard curve). Determine the variance (S^2) for each analyte as follows: $$s^2 = \frac{1}{n-1}\left[\sum_{i=1}^{n}(x_i - \bar{x})^2\right]$$ where x_i = the ith measurement of the variable x and $\bar{x}$ = the average value of x; $$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i$$ Determine the standard deviation (s) for each analyte as follows: $$s = (S^2)^{1/2}$$ Determine the MDL for each analyte as follows: $$MDL = t_{(n-1,\ \alpha = .99)} * (s)$$ Where $t_{(n-1,\ \alpha = .99)}$ is the one-sided t-statistic appropriate for the number of samples used to determine (s), at the 99 percent level.

Table 2
List of Available Appendices
(Not included in this document)

Appendix Number	Appendix Topic
1	Percentage Points of the t Distribution (Reproduced from "Introduction to Statistical Control" by Douglas C. Montgomery)
2	F Distribution Table for alpha = 0.10 (Reproduced from "Introduction to Statistical Control" by Douglas C. Montgomery)
3	MDL, LLR and MQL Summary for Aqueous and Related Matrices
4	MDL, LLR and MQL Summary for Soil and Related Matrices

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The appendices to this SOP are dynamic laboratory tools that may be updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices. The appendices may be available in hardcopy form or as portable document electronic files (pdf).

Appendix 1

Percentage Points of the t Distribution

(From Introduction to Statistical Control by Douglas C. Montgomery)

NOTE: *This appendix is left blank when this SOP is provided for entity external to AML.*

Appendix 2

F Distribution Table for alpha =0.10

(From Introduction to Statistical Control by Douglas C. Montgomery)

NOTE: *This appendix is left blank when this SOP is provided for entity external to AML.*

Appendix 3

MDL, LLR and MQL Summary for Aqueous and Related Matrices

(Groundwater, Wastewater, Drinking water, etc.)

NOTE: *This appendix is left blank when this SOP is provided for entities external to AML. Current information is updated as often as necessary and provided to clients, regulators and/or auditors upon approval by AML management*.

Appendix 4

MDL, LLR and MQL Summary for Soil and Related Matrices

(Soil, sediment, Waste, etc.)

NOTE: *This appendix is left blank when this SOP is provided for entities external to AML. Current information is updated as often as necessary and provided to clients, regulators and/or auditors upon approval by AML management*.

STANDARD OPERATING PROCEDURES

No. Q-ProjectMgmt-RD

Project Management

October 31, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This Analytical Management Laboratories (AML) standard operating procedure (SOP) is designed to provide uniformity and guidance for Project Coordination within the laboratory. AML employees should adhere to the procedures set forth in this document so that projects can be systematically coordinated.

2.0 SUMMARY OF METHOD

2.1 When a project is in the planning stage and/or in the contracting stage, a client representative normally contacts the laboratory for laboratory capabilities, method performance data and cost for analysis. A cost estimate is prepared by one of the AML personnel: Marketing Manager, Project Manager or Operations Manager. Method performance data (MDLs, control limits, etc.) and operating procedures (QAM, SOPs, etc.), which are normally off-the-shelf materials prepared by the QA department are also provided to clients. The AML employee that provides the initial information remains the client's point of contact (POC) or the Project Manager for the duration of the project unless other personnel are assigned.

2.2 When samples begin arriving, the project information is verified and the samples are logged into the LIMS. Problems with the samples are documented and the appropriate client personnel are contacted by the AML Sample Custodian and/or the AML Project Manager to resolve problems. When all the analyses are complete, the data are compiled and a report is delivered to the client.

3.0 INTERFERENCES

3.1 Three-way contracting with sample reports delivered to one company and billing to a different company may cause problems and it should be avoided.

3.2 The most significant problem is multiple points of contact for the client. Whenever possible, a single point of contact should be encouraged to keep confusion to a minimum.

3.3 Verbal client instructions may conflict with written documents or project plans. Deviations should be documented in written internal records and provided to the client either as part of the report or as a stand-alone document.

4.0 APPARATUS AND MATERIALS

4.1 Personal Computers.
4.2 Laboratory Information Management System
4.3 AML Forms (Chain of Custody, Cooler Receipt Form, invoice, etc.)
4.4 Telephone
4.5 Facsimile transmission machine (FAX)

4.6 Electronic Mail (Email)
4.7 Writing pads, pen, pencil, etc.

5.0 REAGENTS

Not Applicable.

6.0 STANDARDS

Not Applicable.

7.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Not strictly applicable. However, AML Project Manager should coordinate support provided by the laboratory for sample collection. This includes providing containers, preservatives, coolers, packaging materials, custody seals, etc. See Section 13, References for AML SOP on this subject.

8.0 HEALTH AND SAFETY

Not Applicable. See Section 13, References for AML SOP on this subject.

9.0 WASTE DISPOSAL

Not Applicable. See Section 13, References for AML SOP on this subject.

10.0 PROCEDURE

10.1 **Project Initiation -** When a project is in the planning stage and/or in the contracting stage, a client representative normally contacts the laboratory for laboratory capabilities, method performance data and cost for analysis. A cost proposal is prepared and submitted to the prospective client by one of the AML personnel: Marketing Manager, QA Director, Project Manager, Operations Manager, or Laboratory Director. The AML employee that provides the initial information remains the AML Project Manager and the client's primary point of contact (POC) for the duration of the project unless other personnel are assigned.

10.2 **Laboratory Information -** The client may request additional information about the laboratory such as the following: statement of qualifications (SOQ), quality assurance manual (QAM) or plan (QAP), and copies of certificates from the State (e.g. Kansas, NELAC, etc.) and/or federal agencies (e.g. USACE). These are normally off-the-shelf materials prepared by the QA department. These may be submitted during project initiation. The QA Manger and/or the QA Director should be involved in this process.

10.3 **Method Information -** The client may request method performance data such as method detection limits (MDLs), control charts, technical standard operating procedures (SOPs), etc. See section 13 for a list of AML SOPs that deal with these subjects. The AML training SOP contains a comprehensive list of current AML SOPs. These are normally off-the-shelf materials prepared by the QA

department are also provided to clients. Due to the size and proprietary nature of these documents these cannot be provided to "potential clients". They can only be provided for use on specific projects. The QA Manger and/or the QA Director should be involved in this process.

10.4 **Project Scheduling -** The AML project manager will obtain sampling dates and sampling schedule from the client contact. Client contacts may include Client Project Chemist, Client Project Geologist, and/or Client Project Manager. The AML project manager should provide a "heads-up" to laboratory personnel through internal Email and/or verbal discussions. The Operations Manager and the QA Manger should be involved in this process.

10.5 **Sample Receipt, Login, and Tracking -** The AML Sample Custodian is responsible for interfacing with client contacts (See Section 10.4) including client field personnel to keep them informed of problems that might have developed during sample collection and shipment. See Section 13, References for AML SOP on this subject for specific items that should be completed to keep the client informed. The laboratory personnel are informed about the required analysis through electronic laboratory information management system (LIMS) and physical systems such as tracking boards, assignment sheets, etc. The AML Project Manager should be involved in this process.

10.6 **Sample Analysis -** The AML Operations Manager tasks the laboratory personnel employee to complete analysis in accordance with applicable AML SOPs and/or client specified procedures within technical holding times and project deadlines. See Section 13, References for AML SOP on this subject.

10.7 **Data Reduction, Review, Verification and Reporting -** Upon completion of the analyses, the analysts prepare sample reports and quality control (QC) reports in accordance with project requirements. These reports and the accompanying raw data are reviewed by the analysts, team leader and the operations manager and/or QA Manager. See Section 13, References for AML SOP on this subject. The Operations Manager and/or the QA Manager will assemble the final report, perform a final review, and develop the case narrative. The LIMS Manger is responsible for providing electronic data deliverables (EDD) that match hardcopy reports. Reports are sent by courier, US mail or hand-delivered by the Project Manager for local projects.

10.8 **Project Billing** - When a report is ready for submission to the client, an invoice and/or a cost estimate (e.g. USACE contract) is prepared and submitted simultaneously. The Operations Manager (or other assigned clerical personnel) is responsible for project billing.

10.9 **Sample Disposal** - The samples are disposed off in accordance with AML SOP (See Section 13) on this subject. The AML Sample Custodian is responsible for sample disposal.

10.10 **Project Data Storage and Project Follow-up -** The LIMS Manager is responsible for data management in all forms (paper and electronic). See Section 13, References for AML SOP on this subject. The AML Project Manager, Laboratory Director and/or the QA Director are responsible for follow-up, which involves contacting the client and fixing any problems that they may have on the reports.

11.0 QUALITY CONTROL

11.1 Only appropriately trained personnel can set up LIMS for client projects.

11.2 Copies of the cost estimate or proposal and the invoice for completed projects should be kept on file along with a record of whom it was faxed to and when.

11.3 All original paperwork (e.g., COC form, cooler Receipt Form, work orders, any handwritten notes, shipping documents) associated with the receipt of samples should be maintained in the project file.

12.0 DATA VERIFICATION AND VALIDATION

Not Applicable. See Section 13, References for AML SOP on this subject.

13.0 REFERENCES

13.1 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 2001.

13.2 EM 200-1-2, Technical Project Planning (TPP) Process, US Army Corps of Engineers, August 31, 1998.

13.3 EM 200-1-6, Chemical Quality Assurance, US Army Corps of Engineers, October 10, 1997.

13.4 ER 1110-1-263, Chemical Data Quality Management for Hazardous, Toxic, Radioactive Waste Remedial Activities, US Army Corps of Engineers, April 30, 1998.

13.5 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 1, Quality Control, December 1996.

13.6 AML Standard Operating Procedures, Q-login-RE, Sample Receiving, Login, and Tracking.

13.7 AML Standard Operating Procedures, Q-MDL-RC, Instrument detection limits (IDL), method detection limits (MDL), reporting limits (RL), method quantitation limits (MQL), and initial demonstration of performance (IDP).

13.8 AML Standard Operating Procedures, Q-CCGM-RB, Generation and maintenance of control charts.

13.9 AML Standard Operating Procedures, Q-SOPs-RB, Preparation of standard operating procedures (SOPs).

13.10 AML Standard Operating Procedures, Q-Training-RA, Technical training.

13.11 AML Standard Operating Procedures, Q-Audit-RB, Laboratory certification and internal quality assurance audits.

13.12 AML Standard Operating Procedures, Q-DataMgmt-RB, Data and information storage and retrieval procedures.

13.13 AML Standard Operating Procedures, Q-DVP-RB, Data reduction, review, verification, and reporting procedures.

13.14 AML Standard Operating Procedures, Q-WasteMgmt-RB, Waste management and disposal including sample disposal.

STANDARD OPERATING PROCEDURES

No. Q-SampleMgmt-RC

SAMPLE RECEIVING, LOG-IN, STORAGE, TRACKING, AND MANAGEMENT PROCEDURES

October 11, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan Date
QA Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This Analytical Management Laboratories (AML) standard operating procedure (SOP) is intended to provide guidance on procedures to be employed for receipt, log-in and tracking of samples received by the laboratory for analysis. This procedure is based on guidance provided in a number of documents such as EPA SW-846 and US Army Corps of Engineers (USACE) guidance documents. See Section 11 for a list of referenced documents.

1.2 This SOP describes procedures, which are based on using a state of the art Horizon Chemware laboratory information management system (LIMS) and computer networks for communication within the laboratory facility and with clients that are external to the facility. New users of this SOP should obtain appropriate training from the information technology (IT) specialist.

1.3 This SOP provides guidance for sample custody, sample receipt, inspection, log-in, and tracking of samples received by AML.

2.0 SUPPLIES EQUIPMENT AND REAGENTS

2.1 Supplies – A steady supply of various sample containers, coolers of various sizes, packaging materials, sample labels, custody seals for the bottles, custody seal for coolers, tapes, and fasteners are needed to provide kits for field sampling.

- 2.1.1 Containers – see Appendix 6 for the type of containers that are used for sampling.
- 2.1.2 Labels – see AML QAP, Chapter 7 for details
- 2.1.3 Custody seals – see AML QAP, Chapter 7 for details

2.2 Equipment

- 2.2.1 Thermometer for checking cooler temperatures – Electronic thermometer with a digital readout to 0.1°C, accuracy of ± 0.5°C, and temperature range of -10°C to 50°C. Calibrate the electronic thermometer quarterly in accordance with the AML SOP on Temperature.
- 2.2.2 Analytical Balance, Center Instrument Company, Model ACA-100 with a digital readout to 0.0001 g, with an accuracy of 0.001 g in the range of interest (10 to 50 g).
- 2.2.3 pH papers of various ranges (e.g. 0-14, 4-10, 0-4, and 10-14) that are commercially available.

2.3 Reagents

- 2.3.1 Reagent Water (Type I for preparing trip blanks)
- 2.3.2 Preservatives – Use ACS Reagent grade chemicals for preserving samples (see Appendix 6 for a list of preservatives) when pre-preserved containers are not purchased from a suitable vendor (e.g. QEC). See AML SOP on standards for documenting the

preparation of preservatives, which includes the assigning of reagent lot numbers for each reagent used in sample preservation. Record the starting and ending date in the logbook for each reagent.

2.3.2.1 Hydrochloric acid, from EMD
2.3.2.2 Nitric acid, from EMD
2.3.2.3 Sulfuric Acid, from EMD
2.3.2.4 Sodium hydroxide, from VWR Scientific
2.3.2.5 Sodium bisulfate, from EMD
2.3.2.6 Purge and trap grade methanol, from EMD

3.0 SAMPLE CUSTODY

3.1 A sample is defined as physical evidence collected from a facility, site or the environment. The primary objective of sample custody is to provide accurate, verified, and traceable records of sample possession and handling from sample container shipment through laboratory receipt and sample disposition.

3.1 Custody Definitions: Sample Custody is defined as all records and documentation that is required to trace a sample from point of origin through disposal after analysis. Chain-of-Custody (COC) is formal written records of all events associated with custody of a specific sample. A sample is in custody if

- 3.1.1 It is in the field investigator's or the transferee's actual possession; or
- 3.1.2 It is in the field investigator's or the transferee's view, after being in his/her physical possession; or
- 3.1.3 It was in the field investigator's or the transferee's physical possession and then he/she secured it to prevent tampering; or
- 3.1.4 It is placed in a designated secure area.

4 SAMPLE RECEIPT

4.1 All samples received in the laboratory are initially handled and processed by the sample custodian or his/her designee. A COC record form (Appendix-1) originating from either AM Lab or the client will accompany most samples received by the laboratory. If requested, the COC will start when pre-cleaned containers are shipped to the field from the laboratory and the COC form will be signed by the sample custodian as the sample containers are relinquished to the client. For samples received without a COC record, the laboratory will originate COC from the time of receipt. This form must be filled out completely and signed in the "relinquished by" section by the person delivering the samples.

4.2 Upon receipt of the samples, AML Sample Custodian will open the cooler or the package in a designated area, including a fume hood, if breakage is suspected. After verifying that the information on the sample containers and information about samples on the COC agree, the sample custodian will sign the COC and document the date and time received. Any discrepancies will be

noted either on the COC and/or the Cooler Receipt Record Form (Appendix-2). Samples received in a single shipment will be assigned a "Work Order (WO) Number", which will be used to track this group of samples. The maximum number of samples in a single AML WO number is 99. For some projects (e.g. EPA), large number (>20) of samples received in a single shipment may need to be broken up into "Sample Delivery Groups" (SDG), each containing twenty samples or less. Several AML WO numbers may need to be assigned in this case for a single sample shipment. The client should be encouraged to submit samples in separate groups of 20 or less to minimize confusion.

4.3 The AML Sample Custodian will assign a Manila folder for each WO. The original COC form, traffic records (FedEx receipts, etc., if any) and other accompanying field documentation (See Appendix-3) will be kept in this folder. Laboratory sample numbers will be assigned to each sample received on the original COC and a copy of the completed COC will be faxed to the client as an acknowledgment of receipt of samples. All entries on the COC record must be signed in ink or typewritten. Any errors should be corrected by drawing a single line through the error, signing and dating the correction.

4.4 The temperature of the samples upon receipt is checked either by inserting the thermometer into the "temperature blank" and recording the temperature, or placing the thermometer into the cooler and closing the lid for two minutes. The temperature is then checked and recorded on the chain of custody and/or the Cooler Receipt Form. If the temperature of the samples is not in the 0 ^{o}C to 6°C range, the client project manager is notified.

4.5 Aqueous samples (except VOA vials) are checked for field preservation by using a pH test strip and the results are recorded on the internal sample tracking form. Perform the pH check and appropriate corrective action as described below:

4.5.1 Using a narrow range pH indicating paper (e.g. 0 to 4 and 10 to 14 or other narrower ranges as necessary for more accurate measurements) record the pH of the sample. This is accomplished by dipping the tip of a clean Pasteur pipette into the sample and placing the droplets of water on the pH paper. Never dip the pH paper into the water sample. Record the outliers on the sample receival report.

4.5.2 If a bottle does not contain the proper preservative for the tests required, notify the AML Project Manager and/or the Client Project Manager.

4.5.3 With the project manager's approval, add an additional quantity of preservatives to previously preserved samples, as needed.

4.5.4 With the project manager's approval, add an appropriate quantity of preservatives to previously un-preserved samples, if required.

4.5.5 Samples for dissolved metals analysis that were collected unpreserved may need to be filtered using 0.45-µ filters and preserved with nitric acid immediately after sample receipt.

4.5.6 NOTE: VOA Vials are never to be opened by anyone other than the analyst, strictly when necessary. Most of these sample vials for both water and soil samples are seldom opened by anyone after sample collection. Do no check the pH of the VOA samples. Check the COC for information on the preservative used for these samples.

4.6 AML policy dictates that all samples sent to the laboratory for analysis are "accepted" by the laboratory and assigned a unique number for tracking

purposes. If samples are received at the lab in broken or leaking containers, improperly preserved, requiring analytical testing not performed by the lab, etc., the samples are logged into the system and the client or project manager is contacted before any analysis begins. Any problems of this nature are clearly documented on the COC form and/or the cooler receipt form along with any corrective action taken.

4.7 The following information should be included on the COC form and/or the Cooler Receipt Form as applicable:

- Date/time of sample receipt
- Courier used by the sample shipper including shipping records, if any
- Name and signature of person delivering the samples (if not sent through a courier company)
- Name and signature of person receiving the samples (sample custodian)
- Name and signature of person relinquishing the samples.
- AML Work Order Number
- Customer Name, Address, Point of Contact
- Customer phone number, fax number and Email address
- Project name
- Customer Sample numbers on COC for each sample collected
- Date/Time of sample collection
- Customer sample identifiers on sample vials for each sample collected
- Sample matrix identifiers for each sample collected
- Due date - based on holding times or client request whichever is sooner.
- Tests required and test method numbers
- Condition of the shipping container
- Condition of the sample bottles
- Appropriate preservation of samples
- Presence or absence of coolants (ice bags, etc.)
- Presence or absence of traffic reports
- Verification of agreement or non-agreement of information on shipping documents.
- Sample tag identification numbers cross-referenced to the client sample numbers
- Presence/absence of custody seals on shipping container(s)
- Discrepancies, if any

5 SAMPLE NUMBERING and LOG-IN

5.1 During the log-in procedure, samples are assigned unique AML sample numbers, which are used exclusively to identify and track samples within the laboratory. Two-digit numbers (01 to 99), which follow the WO numbers, are currently used by the laboratory. If a sample consists of more than one container, each sample is identified with the same unique sample number with a unique suffix (e.g. A, B, and C for the three VOC vials, D and E for the GRO vials, F and G for the two SVOCs 1-L bottles, etc.) for each of the containers that constitute one sample. The sample custodian is responsible for assigning sample numbers and container suffixes.

5.2 Samples designated for matrix spike (MS) and matrix spike duplicate (MSD) analyses are assigned the same AML sample number. The LIMS will

designate this sample for triplicate analysis, once without spike, and twice with the spike.

5.3 Separate A-codes exist in the LIMS for each sample preparation procedure and instrumental analysis procedure. These codes are entered for each sample that corresponds to the analyses requested by the client on COC or other documents. See Appendix-4 for a list of A-codes.

5.4 Special requirements such as project-specific MDLs, control limits, special spiking levels, etc. are entered into the LIMS as comments, which are available to the laboratory analysts.

5.5 Prepare a log-in report as shown in Appendix-5. This report may be either faxed or emailed to the client in addition to other documents such as the completed COC and Cooler Receipt Forms.

5.6 The LIMS provides internal notification for the entire laboratory.

5.7 Samples require preparation and analysis within required technical holding times. See Appendix-6 for a summary of the containers, preservatives used and applicable holding times.

6 SAMPLE STORAGE

6.1 After log-in is completed, samples are stored by in designated areas by the sample custodian. These areas are:

Aqueous VOCs Samples	=	Samples stored in a dedicated upright refrigerator located in the VOA laboratory
Soil VOCs (5035) Samples	=	Samples stored in a dedicated freezer located in the sample log-in area
All other samples	=	Walk-in cold storage refrigerator in the sample log-in area

6.2 The sample custodian is responsible for monitoring the temperature of sample storage and other temperature-controlled storage areas. See appropriate AML SOPs that deal with these procedures.

6.3 Analytical processing begins as the appropriate analytical teams are notified of the sample receipt through LIMS.

7 SAMPLE TRACKING

7.1 Sample tracking is done using the LIMS. Samples that require extraction or digestion appear as backlogs for each A-code on the LIMS. As the extraction is completed and the preparation QC batch information entered into the LIMS, sample extracts become backlogs for the instrumental analyst. As the instrumental analysis completed and the analysis QC batch information entered into the LIMS, sample results become backlogs for report preparation.

7.2 Due to the relatively small size of the laboratory, there is no internal tracking of sample containers within the laboratory. Larger facilities may have sign-up sheets, which must be completed, whenever samples or sample containers are either removed from or returned to the sample storage area. The initials, date, time, and the quantity of sample removed are commonly tracked. AML used

such a system in the past, and it reserves the right to use it again for special projects that may require such tracking.

7.3 AML uses a simple system for internal tracking of samples. Analysts should remove sample quantities only from containers that are marked for the respective analysis that they perform. Analysts should notify their supervisor and/or the laboratory project Manager immediately if they suspect that sample quantities are insufficient to perform all the required tests.

8 SAMPLE AND LABORATORY SECURITY

8.1 Sample security and laboratory security are maintained by keeping all outside access doors, except the front door, locked at all times. A laboratory staff should be on duty to keep vigil on the front door. Visitors are required to register at the front desk and are escorted by laboratory personnel while in the facility.

8.2 The sample storage refrigerators and/or freezers are not locked. AML used such a system in the past, and it reserves the right to use it again for special projects that may require additional security.

9 INTER LABORATORY CHAIN OF CUSTODY

9.1 If it is necessary to transfer samples and/or extracts to another laboratory, a completed, signed chain-of-custody record must be included with the transfer. See Appendix 1 for a copy of the COC form.

9.2 If sample extracts are being transferred to another laboratory for instrumental analysis, it is also necessary to include a copy of the sample preparation log. Contact the applicable Team Leader for necessary information.

10 SAMPLE DISPOSAL AND OVERSEAS SAMPLES

10.1 Overseas samples, including samples from US territories such as Hawaii, Puerto Rico, Guam, etc. are to be treated as any other sample with the exception that a sticker noting the origin of these samples are to be placed on all of the sample containers.

10.2 Overseas samples require autoclaving prior their disposal or additional procedures that are described on the permit (Appendix-7). These samples should be disposed of in accordance with their permits and in accordance with the AML SOP on sample disposal.

11 REFERENCES

11.1 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Revision 4, December 1996.

11.2 Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, EPA, 40 CFR Part 136, October 1984.

11.3 Methods for the Determination of Organic Compounds in Drinking Water, EPA/600/4-88-039, July 1991.

11.4 EM 200-1-1, Validation of Analytical Chemistry Laboratories, US Army Corps of Engineers. July 1, 1994.

11.5 EM 200-1-2, Technical Project Planning (TPP) Process, US Army Corps of Engineers, August 31, 1998.

11.6 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, September 1, 1994. Appendix I.
11.7 Horizon LIMS Users Manual, AML, 1999.
11.8 AML Standard Operating Procedures, Q-WMADISD-RA.
11.9 AML Standard Operating Procedures, Q-Weights-RA.
11.10 AML Standard Operating Procedures, Q-Temperature-RA.
11.11 AML Standard Operating Procedures, Q-Volumes-RA.
11.12 AML Standard Operating Procedures, Q-CCGM-RA.
11.13 AML Standard Operating Procedures, Q-MDL-RA.

Table 1
List of Available Appendices
(Not included in this document)

Appendix Number	Appendix Topic
1	AML Chain of Custody and Analysis Request Form
2	AML Cooler Receipt Form
3	Traffic Reports
4	List of AML LIMS A-Codes
5	AML LIMS Log-in Report
6	Containers, Preservatives and Holding Times
7	Overseas Soil Permit
8	AML Packing Slip for Sample Containers

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices. The appendices may be available in hardcopy form or as portable document electronic files (pdf).

STANDARD OPERATING PROCEDURES

No. Q-Software-RD

SOFTWARE ACQUISITION, VALDATION, AND TRAINING

October 31, 2005

APPROVED:

_____ _____
Laboratory Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This Analytical Management Laboratories (AML) standard operating procedure (SOP) is designed to provide guidance for the training, use and validation of software acquired or developed by AML.

2.0 SUMMARY OF METHOD

2.1 When new software is acquired or developed by the laboratory, it should be validated to verify that it automates the performance of tasks in accordance with stated procedures. Routine use of software requires an understanding of it capabilities and training in its use to complete applications.

2.2 Software developed by the laboratory is validated by the Information Technology Specialist (ITS). Software acquired by the laboratory is validated by the analyst who will be using it routinely. The QA staff (QAS) will provide assistance in its implementation.

3.0 INTERFERENCES

3.1 Software from different companies may not work smoothly together. Debugging should be performed by ITS prior to their use in the facility.

3.2 Algorithms used in acquired software may differ from those specified in commonly used in methods. Appropriate modification may be necessary prior to their routine use.

4.0 APPARATUS AND MATERIALS

4.1 Personal Computers.

4.2 Laboratory Information Management System

4.3 Software

5.0 REAGENTS

Not Applicable.

6.0 STANDARDS

Not Applicable.

7.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Not Applicable.

8.0 HEALTH AND SAFETY

Not Applicable.

9.0 WASTE DISPOSAL

Not Applicable.

10.0 PROCEDURE

10.1 **Software Training -** See AML SOP on training (Section 13) for training provided to new employees. Training in the use of specialized software should be completed by attending classes provided by the vendor. The employees should address specific applications to the instructor during these classes for compliance of the software with the intended application. Online training and tutorials included in software should supplement training courses that may be offered by the vendor. Common software may not need extensive training in completing simple tasks.

10.2 **Software Use -** The analysts should make themselves familiar with software capabilities and in their routine operation. See AML SOP on data management (section 13) for details. The ITS should be consulted when instruments that employ new software are interfaced to the Laboratory Information System (LIMS).

10.3 **Software Validation -** The software may complete calculations using mathematical equations and functions that are either identical to or quite similar to those employed in calculations performed by environmental laboratories. These should be verified by performing calculation independently using alternate software (e.g. Excel) or electronic calculators. An initial verification should be performed before using software in the analysis of client samples. Continuing verification is not necessary. Contact QA Staff for more details.

10.4 **Software Custody** - The ITS is responsible for the safekeeping of the original licensed software. The IT Specialist is the only AML authorized employee that can install new software in any of the AML-owned computers. Pirated software should not be installed in AML computers.

11.0 QUALITY CONTROL

11.1 Only appropriately trained personnel can set up new software. This includes the IT Specialist and other employees trained in specialty software.

11.2 Instrument printouts and copies of the software validation results should be kept on file for potential review by laboratory auditors.

12.0 DATA VERIFICATION AND VALIDATION

Not Applicable. See Section 13, References for AML SOP on this subject.

13.0 **REFERENCES**

13.1AML Standard Operating Procedures, Q-Training-RC, Technical training.

13.2AML Standard Operating Procedures, Q-DataMgmt-RD, Data and information storage and retrieval procedures.

STANDARD OPERATING PROCEDURES

No. Q-Sonicator-RE

SONICATOR TUNING

October 31, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes Analytical Management Laboratories (AML) procedures for the adjustment and tuning of the sonic disruptor(s) that are used in sample preparation procedures.

2.0 SUMMARY OF METHOD

Tuning optimizes performance and insures maximum transfer of energy by matching the frequency of the power supply to that of the converter/probe assembly. The piezoelectric crystal within the converter is part of the circuitry, which controls the frequency at 20-kHz. Any changes in the crystal's capacitance such as the probe, the probe temperature, etc. will cause the equipment to operate in an out-of-tune condition. Procedures described in this SOP include adjustments that are made to the power supply while the power supply is attached to the probe, which has reached operating temperature.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

Tekmar Model TM500 Sonic Disruptor with 500-watt power and pulsing capabilities.
Tekmar Model V1A 3/4" Horn (probe).

5.0 REAGENTS

None

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Not applicable.

7.0 PROCEDURE

7.1 Frequency: The power supply should be tuned whenever the following changes are made:

7.1.1 Whenever a new probe or accessory is used
7.1.2 To compensate for the frequency variation caused by cavitation erosion.
7.1.3 Following 10-minutes of continuous operation
7.1.4 Whenever sample temperature is significantly higher or lower (±10° C) than the room temperature.

7.1.5 Whenever the environment of the ultrasonic processor is changed (e.g. from very cold to very hot.)
7.1.6 Tune once a week or as often as necessary based on criteria shown above.

7.2 Power Supply Description: The power supply has a red indicator light that comes on when the unit is turned on. IT is equipped with two toggle switches that control ON/OFF/Tune and Pulsed/Continuous operations. It has a timer (1-15 min.), analog power readout (0 to 100) with a needle, a duty cycle potentiometer (10 to 80) and an output control potentiometer (1 to 10). It has a tune knob potentiometer at the back of the power supply.

7.3 Procedure: Remove probe from the sample. Set pulser to off position. Depress the tune switch and rotate the tuner knob (at the back of the machine) clockwise or counterclockwise until a minimum (usually less than 5) is obtained on the power monitor (at the front of the machine). Power monitor reading should be less than 20. Check for loose probe if minimum cannot be obtained. Service the power supply or horn if necessary.

8.0 REFERENCES

8.1 Tekmar Instrument Manual.
8.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3500B, Organic Extraction and Sample Preparation, December 1996.
8.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 3550B, Ultrasonic Extraction, December 1996.

STANDARD OPERATING PROCEDURES

No. Q-SOPs-RE

PREPARATION, DISTRIBUTION, AND CONTROL OF STANDARD OPERATING PROCEDURE (SOP) DOCUMENTS

October 11, 2005

APPROVED:

_____ _____
Laboratory Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) provides guidance for the development of technical and administrative SOP documents that are intended to provide more detailed guidance for various technical, quality control, administrative, and management procedures employed at Analytical Management Laboratories (AML).

1.1 AML has sole discretion and rights over the AML SOP content. Although comments and suggestions from clients and regulatory agencies may be considered while making revisions to the SOPs, project specific documents (e.g. Quality Assurance Project Plans or QAPP) should describe required modifications and/or changes that may be required when performing specific tests for specific projects. The written SOPs should not be modified to accommodate specific clients or projects.

1.2 This SOP is based on the EPA document, "Guidance for the Preparation of Standard Operating Procedures (SOPs) for Quality-Related Documents, EPA QA/G-6". This SOP and other SOPs developed with or without using this guidance document are "living documents".

1.3 While the SOPs are intended to provide structure and organization necessary to perform tasks in accordance with applicable standard requirements, AML is aware of client requirements (e.g. rapid turnaround times for analysis that are not sufficient to perform applicable corrective action, method modifications, etc.) that will supersede AML SOP provisions. Therefore, AML reserves the right to revise and change this document, and to deviate from written procedures to accomplish project objectives that have been communicated in both written and verbal forms.

1.4 Procedures described in the AML SOPs and actual procedures followed should be aimed at accomplishing the following: to nurture and/or accommodate employee-creativity, operational efficiency, personnel and equipment requirements, responsiveness to client requirements, responsiveness to changes in the marketplace, and responsiveness to regulatory changes.

2.0 SUMMARY OF PROCEDURE

The AML SOPs will be developed and organized in a manner similar to the EPA SW-846 methods with additional guidance from outside laboratory auditors. AML SOPs will contain facility-specific information and procedures that are unique to AML in addition to generic information that is derived from referenced methods. Proprietary information, which may include facility-specific information, should be included in appendices. Circulation should be limited for information contained in appendices.

3.0 INTERFERENCES

Not Applicable.

4.0 APPARATUS AND MATERIALS

Not Applicable.

5.0 REAGENTS

Not Applicable.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Not Applicable.

7.0 HEALTH AND SAFETY

Not applicable other than watching out for paper cuts.

8.0 WASTE DISPOSAL

Not applicable.

9.0 PROCEDURE

9.1 AML SOPs should have the following sections for technical SOPs:

Section	Section Title
-	Cover Page (with signatures)
1	Scope and Application
2	Summary of Procedure (Method or SOP)
3	Interferences
4	Apparatus and Materials
5	Reagents
6	Sample Collection, Preservation and Handling
7	Health and Safety (included per auditor recommendations)
8	Waste Disposal (included per auditor recommendations)
9	Procedures
10	Calculations
11	Quality Control
12	Data Verification and Validation (included per auditor recommendations)
13	References

9.2 Each section should contain facility-specific information and procedures that are unique to AML in addition to generic information that is derived from referenced methods. Proprietary information, which may include facility-specific information, should be included in appendices. Circulation of the appendices should be restricted to AML employees and outside auditors.

9.3 Additional sections may be added. Sections may be removed and combined with related sections. For example, Section 9 Procedures may be broken up into additional sections or subsections (e.g. sample preparation, instrument calibration, troubleshooting, sample analysis, etc.). Section 10, "Calculations" may be replaced by or appended to sections dealing with data acquisition, data reduction and data

management. Sections 11 and 12 may be combined if they contain redundant information. The SOP author may exercise professional judgment in making these modifications with approval from either the QA Manager and/or QA Director. Although it is desirable to keep SOPs as self-contained as possible, some sections that are not specific to the technical SOP may be incorporated by reference to information contained in other AML SOPs to keep the size of SOPs manageable.

9.4 The number of SOP sections and their content are likely to change in the immediate future when performance based measurement systems (PBMS) for technical operations are incorporated.

9.5 The SOPs should contain control documentation in headers that should include the facility name, SOP number, date, and page number. Footers that describe the content such as title may be included if the SOP number is not indicative of the SOP content.

9.6 Types of SOPs: Administrative SOPs on topics such as this SOP topic may contain sections (e.g. Sample Collection, Preservation, and Handling) that are "Not Applicable" due to the nature of the subject. These sections may be deleted from administrative SOPs. Additional sections and/or subsections dealing with specialized issues (e.g. definitions, personnel qualifications, etc.) may be included.

9.7 SOP Implementation: Copies of the SOP should be distributed internally within the laboratory to personnel assigned to perform work contained in the SOP. Applicable AML administrative documents should be completed to document SOP training, employee proficiency, etc. The SOP documents, by themselves should not contain signature page (s) for the users and/or reviewers of the SOP.

9.8 SOP Master List: SOPs should be used for internal training. See the current version of the AML SOP on training (See Section 13) for a list of AML SOPs.

9.9 Distribution and Tracking: Use the form in Appendix 1 to track SOP distribution. Maintain a three-ring binder to track SOP revision and distribution. The tracking should include the name of the recipient and their organization. It should include the revision number (RA, RB, etc.) and information on receipt of appendices, Email transfer, etc.

9.10 Review and Updating: The SOPs should be reviewed for accuracy and adequacy and updated annually or at other frequencies based on need. Updating will be required whenever procedural method changes occur. Updating may also be performed in response to client requirements as long as client requirements do not contradict or confuse underlying method requirements.

10.0 CALCULATIONS

Not applicable.

11.0 QUALITY CONTROL

AML SOPs require review and approval by the Author, Laboratory Manager/Operations Manager, and the Quality Assurance Manager/Director. SOPs and operations will also be subject to random and/or periodic audits to assess actual operations in relations to written SOPs.

12.0 DATA VERIFICATION AND VALIDATION

Not Applicable

13.0 REFERENCES

13.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 1, Quality Control, Revision 1, July 1992.
13.2 Guidance for the Preparation of Standard Operating Procedures (SOPs) for Quality-Related Documents, EPA QA-/G-6, EPA/600/R-96/027, November 1995.
13.3 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, September 1, 1994.
13.4 Guide to Method Flexibility and Approval of EPA Water methods, Prepared by Analytical Methods Staff, Engineering and Analysis Division, Office of Science and Technology, Office of Water, U.S. EPA, Washington, DC, December 1996, Chapter 3.
13.5 AML Standard Operating Procedure, Q-Training-RD, Technical Training.
13.6 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001, Appendix I.
13.7 Department of Defense Quality Systems Manual (DoD QSM), Final Version 2, June 2002.

Appendix 1

SOP Distribution Log

(Following Page or Equivalent)

Analytical Management Laboratories
SOP Distribution Log

SOP Number:					
SOP Title:					
Recipient Name	**Recipient Company**	**Revision Number (RA, RB, etc.)**	**Answer Yes (Y) Or No (N)**		
			Hard Copy	**Appen-Dices**	**Email**

STANDARD OPERATING PROCEDURES

No. Q-Standards-RC

ANALYTICAL STANDARDS AND REAGENTS

October 31, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

ANALYTICAL STANDARDS AND REAGENTS

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) describes the procedures that are employed at Analytical Management Laboratories to document reagents and analytical standards that are used in generating environmental analytical data.

1.2 The procedures described in this SOP are used to establish traceability of standards used in the laboratory to the reference standards.

2.0 PURPOSE

2.1 Standards used in instrument calibration, calibration verification, and for spiking blanks, laboratory control samples and matrix spike samples should be traceable to reference standards for legal defensibility of data.

2.2 Procedures described in this SOP are essential for documenting and using information necessary to perform accurate environmental chemical analysis.

3.0 STANDARDS LOG

3.1 Definitions

3.1.1 A ***primary reference standard*** can be defined as a homogenous material with specific properties such as identity, purity, and potency that has been measured and certified by a recognized and qualified organization. They may be a pure chemicals (neat), or standard chemical(s) in a matrix or solution. They are available from sources such as U.S. National Bureau of Standards (NBS), U.S. Phrmacopeial Convention (USP), American Society for Testing and Materials (ASTM), etc. These materials may be quite expensive and they may not be used in everyday operations. Stock standards either derived from or certified against primary reference standards are commonly used in routine operations.

3.1.2 A ***stock standard solution*** is the most concentrated house reference standard. They may be acquired at no cost from government sources, prepared in-house or purchased from commercial vendors. They may be solutions of neat materials at specified concentrations, or mixes designed for specific analytical methods. Certificates of analysis are generally shipped with each labeled vial or ampoule containing the stock solution. These certificates and labels should contain the following information: catalog number and/or lot number, description of the chemical(s) and concentration(s), solvent used and expiry date. Stock solutions prepared in-house should also contain the date and initials of the analyst that prepared the solution.

3.1.3 An ***intermediate standard solution*** is normally prepared in-house from either primary reference standards or stock standard solutions. The source and lot

numbers of all the reagents used and the reference/stock standards used to prepare intermediate standard solutions should be documented for traceability. Logbooks and/or labels should contain the following information: name for the standard, reference number, source and lot number of the parent standards and other reagents, an expiry date and the date and initials of the analyst that prepared the intermediate standard solution. Intermediate standard solutions may be prepared for instrument calibration (ICAL, CALV) and/or for spiking (LCS, MS, surrogates, etc.) samples.

3.1.4 A ***working standard solution*** is normally prepared in-house from either stock or intermediate standard solutions. The source and lot numbers of all the reagents used and the reference/stock standards used to prepare working standard solutions should be documented for traceability. Logbooks and labels should contain the following information: name for the standard, reference number, source and lot number of the parent standards and other reagents, an expiry date and the date and initials of the analyst that prepared the working standard solution. Working standard solutions are normally prepared for instrument calibration (ICAL, CALV) and/or for spiking (LCS, MS, surrogates, etc.) samples. Working standards may be stored at higher temperatures than intermediate/stock/reference standards for convenience in use. Their expiry dates should be consistent with the stability of the standards and the type of handling.

3.1.5 ***Reagents*** such as acids, base, chemicals and solvents are acquired from commercial sources. Reagents are available in different grades and quality and they are intended for different applications. Certificates of analysis are generally shipped with each shipment of these reagents. The source, lot number, and expiry dates of reagents should be recorded in the standards logbooks and in sample preparation logs.

3.1.6 ***Second Source Standard Solutions*** may be reference, stock, intermediate or working standard solutions, which are used to verify the authenticity of the primary

3.2 General Procedures

3.2.1 ***Certificates of Analysis*** (Appendix 1) for reference/stock standards and reagents that are purchased from outside sources should be maintained in binders and made available for audit purposes. Copies of the certificates may be made and pasted in in-house Standard and Reagent Logs. Separate binders should be used for the following: 1. Organic standards, 2. Inorganic standards, 3. Organic solvents, and 4. Inorganic reagents such as acids. The Sample Custodian, who is also responsible for shipping and receiving, maintains these binders.

3.2.2 ***Standard Preparation Log*** entries (Appendix 2) are made to document the preparation of all in-house standards, which will include stock, intermediate and working standards. An example is shown in Appendix 2. Logs similar to those included in Appendix 2 are used for metals analysis. The standard

name and reference number shown for the standard solution on the Standard Preparation Log should be used in sample preparation logs, instrument run logs and related documents for traceability. For example, the ICP-AES instrument printout (Appendix 3) for STD1 contains the standard name (STD1) as the sample name and the reference numbers (M991123-01), book numbers (1) and page numbers (1) in the comments section. Selecting a name for the standard and the reference number (lot number) are left to the analyst's discretion.

3.2.3 ***Expiry Dates*** should be included for standards prepared in-house. Expiry dates should be based on the known stability of the standards and they need not default to the expiry date recommended by the vendor. Commercial vendors typically use short expiry dates to promote additional sales for themselves, which will lead to excessive costs for the acquisition and disposal for the laboratory. Analysts should prepare quantities of standards that are appropriate based on the stability and use rates to minimize waste.

4.0 IMPLEMENTATION

4.1 Team leaders (metals, wet chemistry, organic chemistry) are responsible for maintaining and reviewing the standards log assigned to their section.

4.2 Analysts are responsible for turning in the certificates of analysis to the Sample Custodian as soon as the standards and/or the reagents are received. Analysts can maintain photocopies of the certificates in the standards log. The Sample Custodian is responsible for maintaining the certificates for standards and reagents.

4.3 The QA Manager and/or the Operations Manager are responsible for auditing the logs.

Table 1
List of Available Appendices
(Not included in this document)

Appendix Number	Appendix Topic
1	Certificates of Analysis
2	AML Standard Preparation Log
3	AML Cross-referencing Procedure for Analytical Standards

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices. The appendices may be available in hardcopy form or as portable document electronic files (pdf).

STANDARD OPERATING PROCEDURES

No. Q-Subsampling-RD

LABORATORY SUBSAMPLING FROM SAMPLE CONTAINERS

October 31, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 1.1 This Analytical Management Laboratories (AML) standard operating procedure (SOP) is designed to provide guidance on the representative subsampling of analytical aliquots from environmental samples received in typically sized field containers (e.g., bottles, split spoon liners, VOA vials, etc.).

1.2 This SOP is based on the Missouri River Laboratory SOP (#Q-021-MRL-GL, Ver. 1.0 dated September 6, 1996), ASTM Method D6328-98, and guidelines contained in the EPA SW-846 Manual.

1.3 It should be noted that specific samples might require special techniques due to problematic matrices or project specific requirements.

1.4 The intent is to address the following matrix groups and subgroups:

1.4.1 LIQUIDS

1.4.1.1 Aqueous liquids — surface water, groundwater, TCLP extracts, wastewaters, and leachates (< 1% solids)

1.4.1.2 Nonaqueous liquids — oils

1.4.2 SOLIDS

1.4.2.1 Soil, sediment,

1.4.2.2 Heterogeneous solid substances

1.4.2.3 Concrete, paint chips, ash

1.4.3 MULTIPHASE (LIQUID/SOLIDS) SLUDGES

1.2.3.1Aqueous sludges — liquid sludges, solid sludges, emulsions

1.2.3.2 Nonaqueous sludges

1.5 This procedure is not applicable to samples collected for VOCs analysis, wipe samples, and air samples for which duplicate field samples should be collected in the field.

2.0 SUMMARY OF METHOD

2.1 The subsampling approaches presented in this SOP are based in large part on the most common characteristics and properties found within each matrix group. However, sampling theory cannot replace experience and common sense.

2.2 Bulk samples received at the laboratory must be thoroughly homogenized, if applicable, prior to subsampling into appropriate containers for analysis and/or shipments to another laboratory.

3.0 INTERFERENCES

3.1 The most significant potential problem is the high probability of a subsample taken from a heterogeneous sample or waste being non-representative. Care must be exercised so that the introduction of contamination or potential for cross-contamination from equipment used to manipulate the sample is minimized.

3.2 Appropriate decontamination procedures should be followed on equipment used between each use.

3.3 Recommend implementing the appropriate quality controls to ensure that samples are not contaminated. Aqueous samples containing hydrophobic analytes, e.g., petroleum hydrocarbons should not be subsampled. The entire bottle contents should be used to avoid loss of analytes to container walls or cap.

4.0 APPARATUS AND MATERIALS

4.1 The specific equipment used for subsampling is dependent on the analyses and methods to be performed. Specific methods should be consulted prior to performing these activities.

4.2 Specific equipment should be selected from the options presented below.

- 4.2.1 Spatulas, spoons, scoops, PTFE-coated or stainless steel, various sizes
- 4.2.2 Stirring rods, glass
- 4.2.3 Magnetic stirrers, PTFE-coated magnetic stir bars
- 4.2.4 Laboratory trays, metal, plastic or glass
- 4.2.5 Laboratory beakers, PTFE or glass
- 4.2.6 Class A Pipette, serological and volumetric (TD, to deliver), various sizes
- 4.2.7 Class A Graduate Cylinders (TD to deliver), various sizes up to 2-L
- 4.2.8 Weigh boats, various sizes
- 4.2.9 Laboratory drying oven
- 4.2.10 Balance, top-loader with readability to 1 mg or analytical with readability to 0.1 mg.
- 4.2.11 Laboratory grinders (manual [mortar/pestle] or mechanical)
- 4.2.12 Sieve — the sieves to have available are dependent upon the analyses performed by the lab. For example, Methods 3540 and 3550 require a 1-mm sieve, 8330 a 30 mesh sieve, 1311 a 9.5-mm sieve, etc.
- 4.2.13 Centrifuge - Marathon 3200 (Fisher), Medifuge (Baxter) or equivalent.
- 4.2.14 Separatory Funnel, glass
- 4.2.15 Filtering apparatus — Glass Microanalysis, 47 mm (Fisher catalogue # 09-753-1c) or equivalent.
- 4.2.16 Syringe filters with Teflon filtering discs
- 4.2.17 Syringes, glass, air-tight, various sizes, with Luer-Lok valves
- 4.2.18 Paper towels - Kimwipes, or equivalent
- 4.2.19 Coring device — Use commercially available (USOil) cut-off syringes designed for 5035 sampling or remove injection end of a disposable plastic 10-cc syringe. Remove Luer-Lok end of the syringe with a razor edge knife, creating a smooth clean barrel neck.

5.0 REAGENTS

5.1 Pesticide grade methanol or equivalent
5.2 Non-phosphate detergent such as Liquinox
5.3 Tap water
5.4 Reagent water (see current revision of AML SOP No. Q-Water-RX)

6.0 STANDARDS

Not Applicable.

7.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Not strictly applicable. However, subsamples that may be used to perform multiple tests and/or re-analyses should be preserved in accordance with applicable method specific SOP (e.g. air-dried samples for explosives analysis).

8.0 HEALTH AND SAFETY

8.1 Samples may contain a number of regulated compounds at high concentrations. The toxicity of these compounds present in such complex mixtures has not been established. Gloves and safety glasses should be worn to prevent contact with skin and eyes.

8.2 Sample vials, which are known to contain high concentrations of organic vapors in the headspace, should be opened in a hood to minimize exposure to organic vapors.

8.3 Proper sample disposal practices (see current revision of AML SOP Q-WMADISD-RX) should be followed to minimize exposure to toxic compounds.

8.4 All subsampling activities should be performed using appropriate personal protective equipment protecting the analyst from direct sample contact, potential vapor inhalation, splashes, and sharp objects.

8.5 Whenever possible or necessary, subsampling should be performed in a properly operating laboratory fume hood.

9.0 WASTE DISPOSAL

9.1 For procedures dealing with the disposal of unused environmental samples, and process waste such as sample extracts, see current revision of the AML SOP on waste disposal (Q-WMADISD-RX).

9.2 The best disposal method for samples is to return them to the point of origin, if possible.

10.0 PROCEDURE

The matrices have been grouped to process samples of similar physical nature. Since this is not "hard and fast" division, subsampling routines should always be evaluated on a continuous basis, based upon subsequent analysis to be performed and the intended use of data resulting from sample analysis. Inspect each sample to determine if the sample is a liquid, solid, or a combination (multiphasic). Depending upon the matrix, refer to sections 10.1, 10.2, and/or 10.3, respectively.

10.1 Subsampling Liquids -Evaluate the liquid sample, looking for suspended matter, multiple phases, or any other features that may require specific measures to obtain a representative subsample. If the samples contain no detectable suspended matter, follow guidance presented in section 10.1.1. Samples, which contain

suspended matter, but will be analyzed as a single liquid phase should follow the guidance presented in section 10.1.2. Multiphase samples should follow guidance established in section 10.3.

10.1.1 Aqueous Liquids — Surface Water, Groundwater, TCLP Extracts, Wastewater, and Leachates (< 1.0% solids)

10.1.1.1 Inorganics and Metals (Total Analysis) - Make sure sample lid is securely attached to sample jar. Invert, or shake the sample in an up and down fashion a minimum of 7 times. Remove lid and quickly yet smoothly transfer desired aliquot into an appropriately sized graduated cylinder. This will be dependent on the required accuracy necessary for the measurement as specified in the applicable method. If the required sample volume is small, a serological or volumetric pipette (depending on required accuracy of measurement) may be used to obtain sample. ˜ Transfer the sample from the pipette or graduated cylinder into an appropriate container used to either process the subsample further or to ship to a contract laboratory for analyses. The sample can be transferred directly into the process apparatus or testing vessel, if appropriate (e.g., cyanide distillation unit). If sample results are not affected by volume additions, rinse the delivery glassware with a small volume (1-2 mL) of reagent water approximately 3 times and transfer the rinsate into the container or test vessel, which contains the sample to ensure a quantitative transfer. If the cylinder or pipette used to subsample is to be reused for other samples, thoroughly clean the transfer glassware appropriately.

10.1.1.2 Dissolved Inorganics and Dissolved Metals - When dissolved analytes are required, field filtration followed by appropriate field preservation techniques is optimum. If this is not practical, project personnel may elect to send unpreserved samples to the laboratory for filtration and preservation. The appropriate regulatory authorities should be consulted and concur with this approach. Generally, the only difference between this and 10.1.1 is that the sample is filtered through a 0.45˜m filter before processing the filtrate through the appropriate procedures. The precipitate is discarded. The filtering device needs to be thoroughly decontaminated in between uses as appropriate.

10.1.1.3 Volatile Organics - VOA vials should never be split for analysis by another laboratory. If split samples are required, separate VOA vials must be collected in the field. VOA samples should be brought to room temperature before subsampling for analysis. For subsampling VOA vials, the best alternative is to collect the subsample through the vial septum using a syringe and needle. If this is not possible, use an airtight syringe fitted with a Luer-Lok valve as follows. Open the VOA vial and smoothly fill the syringe by pulling the plunger smoothly and quickly to

minimize loss of volatiles. Invert the syringe, and express any air bubbles out through the Luer-Lok. A 5-ml syringe is large enough to aspirate more than the 5 mL required for a sample. Duplicate analysis can be accomplished by multiple fillings of the 5-ml syringe. If insufficient quantities are provided for multiple analysis, the use of a second (duplicate) sample vial, if available, may be necessary. Adjust the plunger to the desired sample volume and close the Luer-Lok valve. The sample is now ready for insertion into the purge vessel (after all appropriate surrogates or spikes are added, if this is part of the analytical procedure).

10.1.1.4 Semivolatile Organics - It is not recommended to subsample a 1-L aliquot from a 1-Gallon container, for residual analytes may adhere to the container inner surfaces. When 1-L sample containers are used, rinsing the bottle with the appropriate solvent is recommended. Most semivolatile organic analyses begin by extracting a 1-liter subsample, and therefore the following reflects this commonality. Subsample volumes and glassware can be adjusted based on laboratory or project needs. Make sure sample lid is attached securely and shake the sample in an up and down (or end over end) fashion a minimum of 3 times. If a 1-liter bottle is received, mark the volume on the sample bottle. Pour the sample smoothly into the extraction vessel. Rinse the sample bottle 3 times with small volumes of the appropriate organic solvent and transfer these rinsates into the extraction vessel containing the sample. Fill the sample bottle to the mark with tap water, and pour into a graduated cylinder to determine the sample volume. Note: If a bottle greater that 1-liter is received, an aliquot. should be poured into a graduated cylinder and then transferred into the extraction vessel. The appropriate organic solvent is then used to rinse the graduated cylinder.

10.1.2 Liquid Samples with Suspended Matter (˜1.0%). Sampling procedures for liquid samples that contain suspended matter are very similar to the procedures outlined in section 8.1.1 with some minor deviations.

10.1.2.1 Inorganics and Metals (Total Analysis) - Invert, or shake the sample in an up and down fashion a minimum of 7 times, or until the sample appears uniformly mixed. Evaluate whether the rate that the suspended matter settles allows sufficient time to acquire a representative aliquot. If the suspended matter settles slowly, the sample will be shaken repeatedly and an aliquot taken immediately. If the suspended matter settles rapidly, the sample shaken repeatedly and immediately transferred to a large beaker. A magnetic stir bar is added and the sample is magnetically stirred until uniformly mixed. While stirring continues, an aliquot is taken by pipette or other subsampling means. Refer to remaining protocol presented in section 10.1.1.1.

10.1.2.2 Dissolved Inorganics and Dissolved Metals - Due to the physical separation of the suspended matter, refer to the procedures outlined in section 10.1.1.2.

10.1.2.3 Volatile Organics - Recommend allowing the suspended particles to settle and follow procedures outlined in section 10.1.1.3.

10.1.2.4 Semivolatile Organics - Due to the assumption that the entire 1 Liter sample is processed through the preparatory process, refer to the procedures outlined in section 10.1.1.4. In the event that only an aliquot of the sample is desired refer to section 10.1.2.1 for guidance.

10.1.3 Nonaqueous Liquids (e.g. Oils) - The need for mixing of these nonaqueous liquids must be investigated. Verify whether minor amounts of particles are present which would require redistribution into the liquid prior to subsampling. Samples with substantial amounts of separated phases should refer to section 10.1.3.

10.1.3.1 Due to the different viscosities, densities, coating properties, etc., the weight of the subsample should typically be determined, rather than attempting to express the subsample in terms of volume. If it is difficult to volumetrically measure a sample because it adheres to glassware walls, weight must be used.

10.1.3.2 If a specific and accurate weight needs to be aliquoted, a serological pipette is a good option for subsampling. Aspirate the sample, and then carefully transfer the sample into a tared vessel, controlling the addition by finger pressure on the top of the serological pipette.

10.2 Subsampling Solids — Soil, Sediment, Heterogeneous Solid Substances, Concrete, Paint Chips, and Ash. When subsampling these matrices, first examine the particle size distribution. A decision must be made as to whether a representative subsample can be obtained without prior sample manipulation. This may entail mixing, particle size reduction (PSR), or particle size separation (PSS) techniques. The decision depends on whether the sample is homogenous or heterogeneous in nature. This is determined visually and based on the physical attributes of the sample. Questions to be considered at this time include: (a) is there a significant amount of oversized material (be it either naturally occurring [rocks] or artificially introduced material [debris])? (b) Are there obvious soil, chemical, or ash inclusions where indiscriminate subsampling might bias the analytical results or caution against specific protocols (e.g., lead chunks, tar balls, solid (grayish-white) explosives material)? (c) ˜ Does the sample tend to segregate into various size fractions easily? If the answer to each of the above considerations is no, then taking a subsample as described in 10.2.1 will likely yield a representative sample. If any answer was yes, then the sample is likely heterogeneous material, and guidance in Sections 10.2.2 should be observed. SW-846 states "Decant or discard any water layer on a sediment sample." "Discard any foreign objects such as sticks, leaves, and rocks." USACE default procedures include these practices. Another important aspect of subsampling

depends on the method of analysis, and the size of the aliquot being taken. Analysis requiring smaller (1-2 gram) aliquots (metals, explosives, etc.) require special considerations compared to analysis requiring larger (30 grams) subsamples (semivolatile organics). Achieving a representative subsample must consider whether PSR or PSS techniques are required. If PSR or PSS techniques are used, implement the appropriate quality controls to ensure that samples are not contaminated.

10.2.1 Homogeneous Material

10.2.1.1 Inorganics, Metals, or Semivolatile Organics - Even if the material received appears homogeneous, the entire sample should be thoroughly mixed using an inert, non-contaminating spatula or rod. This may be performed within the original sample container, or the material may be transferred onto a laboratory tray or pan and mixing performed there. For cohesive material, the bulk material should first be size reduced using a stiff bladed utensil such that the average size of any clump is approximately pea-sized (approximately 6mm), if this is possible. The material is then mixed by turning the volume of material over a minimum of three times, followed by taking an appropriately sized subsample and transferring this subsample as before into the preparation or test vessel. (An attempt should be made to subsample equivalent aliquots from different areas of the container or tray until the method prescribed amount is achieved.) Some samples may be received in brass liners or sections of brass liners. These samples must first be extruded onto a laboratory tray or pan and mixed as described above.

10.2.1.2 Volatile Organics - The volatility of the target analytes must be considered when taking a subsample. An alternative method to that described below, is to place the field sample directly into an aliquot of pesticide grade methanol (addition of methanol may elevate MDL) or organic-free reagent water preserved with mercuric chloride so that the soil to liquid ratio is approximately 1:1. (Secondary waste generated should be considered before using this approach.) The soil sample must be completely dispersed in liquid prior to VOC analysis of the liquid portion. (This approach must have prior acceptance from the USACE project manager.) Also refer to other USACE technical reports on this subject. The ideal manner in which to take a volatile subsample is to use a coring. device that takes a representation from non-disturbed soil within the container upon being pushed through the sample. If duplicate analysis is required, a separate collocated field sample should ideally be collected. Discard the top layer of the solid sample. Take approximately equal aliquots from several places within the container. Work should be performed quickly so as to minimize the potential loss of volatile constituents.

10.2.2 Heterogeneous Material - If a solid sample is determined to be heterogeneous (see Section 10.2), a decision must be made as to how a subsample is to be taken so project needs are met. This decision must be made in conjunction with the project manager who submitted the samples for analysis. The following options are possible techniques that can be used to produce a representative subsample of a heterogeneous material. Achieving a representative subsample must consider whether PSR or PSS techniques are required. If PSR or PSS techniques are used, implement the appropriate quality controls to ensure that samples are not contaminated.

10.2.2.1Inorganics, Metals, or Semivolatile Organics - PSR should be avoided for semivolatile organic procedures due to the potential loss of more volatile analytes. Other general considerations include contamination that may result from these techniques. If PSR is necessary, mortar and pestle is usually the best alternative for semivolatile methods. Milling is also an effective technique, but samples must be dry or dried, therefore less desirable for semivolatile methods. If PSR techniques are used which require dry samples, they should be dried at room temperature to a constant weight without exposure to direct sunlight. If the sample is amenable to PSR (e.g., grinding or milling), refer to general guidance above and process the entire sample in an appropriate device (appropriate meaning that target analytes are unaffected by the use of the particular grinding apparatus). After PSR has been accomplished, use of an appropriate sieve to check for oversized material. Rework the oversized material. Following PSR/PSS techniques, mix the sample until homogenous and subsample as outlined in 10.2.1.

10.2.2.1.1 OR - If the heterogeneity is due to foreign material or debris, physically separate the foreign material, mix the remaining homogenous sample, and transfer an appropriately homogeneous sample to an appropriately sized aliquot to the tared testing vessel(s).

10.2.2.1.2 OR - Cone and quarter the samples, repeating this procedure until the subsample obtained meets the sample size of the analytical procedure, as per ASTM Method C702, Method B or ASTM Method D6328-98.

10.2.2.1.3 OR - For samples that segregate into size fractions easily, perform a particle size separation (PSS) procedure (e.g., sieving). Determine the percentage of each size fraction. Compose a subsample that takes an appropriate percentage of each fraction.

10.2.2.2 Volatile Organics - Volatile sample subsampling precludes any aggressive mixing or manipulation of original sample. In this case, the project manager must be consulted to determine the most appropriate manner in which to subsample the container. A good alternative is to use methanol as described in Section 10.2.1.2, if this will meet project needs.

10.3 Subsampling Multiphase (Aqueous/Nonaqueous Liquids/Solids) Sludges. This section is meant to address samples considered multiphasic based on physical

characteristics (mixture of solids and liquids). The choice of the procedure for handling multiphasic samples is highly dependent on project needs. There are no specific procedures describing how samples with certain volume distributions of liquid/solid are handled. It is essential that the USACE project manager be consulted before selecting one of the approaches outlined below. If no clear guidance is provided, analyst judgement must be used to decide what portions will be sampled and analyzed. Regardless of the approach used, it must be adequately documented. Three approaches are provided in the following subsections:

10.3.1 Subsampling of samples analyzed as a Single Mixed Phase (as received). This approach will not provide information on the abundance of analytes in the individual phases other than what can be implied by solubility.

10.3.1.1 The sample is mixed sufficiently so as to create a homogenous sample. This is usually assessed on a visual basis. A single analysis will then be performed. The manner in which the sample is mixed is highly dependent on sample consistency and how easily the phases mix. Some samples can simply be shaken, while others will require a spatula or mixing rod, or laboratory blender. If this is necessary, the device used to mix the sample must be noncontaminating, inert, and easily decontaminated. Usually a glass, or Teflon-coated device is appropriate.

10.3.1.2 The sample is then poured, subsampled with a scoop, or transferred by some other physical means into a tared vessel and the weight of sample is recorded. This transfer is dependent upon sample viscosity/consistency. Another consideration is to not allow the sample to resegregate into the phases when aliquoting the sample.

10.3.2 Subsampling of samples analyzed as Separate Phases

10.3.2.1 When the phases of a multiphasic sample are to be tested individually, the phases are separated by physical means (i.e., filtration [either pressurized or non-pressurized], centrifugation, settling, or use of a separatory funnel). The technique used is dependent on items such as laboratory capabilities, sample characteristics, and analytes of interest. If an aliquot (subsample) of a sample is to be phase separated, this aliquot must be representative of the original sample. This means that the solid and liquid ratio of the aliquot needs to be the same as the original sample.

10.3.2.2 To accomplish this, follow the procedures listed in Section 10.3.1, except that the final step is to aliquot the subsample into the device used to accomplish phase separation. Items to consider when performing the phase separation include: Device is non-contaminating. Device is non-absorbent for analytes of interest. Device does not cause the loss of analytes through other means other than noted above.

10.3.2.3 Once the phases are separated, the solid and liquid ratios are recorded. The two portions are transferred into either sample preparation vessels, testing vessels, or into appropriate storage containers for later analysis. If the liquid portion is subsequently subsampled, follow procedures listed in section 10.1. If the solid portion is subsequently subsampled, follow the solids handling procedures described in section 10.2.

10.3.3 Subsampling of samples analyzed as Separated Phases and only select phases are desired - This approach requires the separation of the phases and the analysis of only those phases of interest. For example, it may be decided that a phase that constitutes only 0-20% of a total sample may be discarded. Again, this approach must be discussed with the client project manager.

10.3.3.1 Separate the phases as described in Section 10.3.2.1.

10.3.3.2 Discard those phases which are not of interest.

10.3.3.3 Transfer portions of the phase(s) to be analyzed into appropriate vessels as described in Section 10.3.2.3.

10.3.4 When phases are separated and analyzed separately, the final concentration for the total sample must be calculated. The final analyte concentration is calculated as follows:

$$\text{Final Analyte Concentration} = \frac{(V_1 * C_1) + (V_2 * C_2)}{(V_1 + V_2)}$$

where

V_1 = Volume (mL) or weight (g) of the first phase
C_1 = Concentration of analyte in first phase
V_2 = Volume (mL) or weight (g) of second phase
C_1 = Concentration of analyte in second phase
The units for concentration, weight and/or volume should be identical for the two phases.

11.0 QUALITY CONTROL

11.1 When processing samples, be sure to fully document the actual procedure used (describing the manipulations as completely as possible), and any irregularities or observations made into the appropriate log book, or bench sheet.

11.2 Check to make sure subsample weights/volumes have been properly recorded in sample preparation log books.

11.3 Implement the appropriate quality control samples to allow an assessment of the potential cross-contamination (blanks), and overall effectiveness of the subsampling techniques (duplicates).

12.0 DATA VERIFICATION AND VALIDATION

12.1 If ineffective subsampling is indicated, the initial subsampling procedure used will be reevaluated. If a more effective method is available, and the holding times are not exceeded, the sample will be resubsampled using the alternative technique, and the analysis repeated.

13.0 **REFERENCES**

13.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, , December 1996.

13.2 Kratochvil, B, and Taylor, J.K., Sampling for Chemical Analysis, Analytical Chemistry, American Chemical Society, July 1981, pp. 924-938.

13.3 Lewis, T.E., et.al., Soil Sampling and Analysis for Volatile Organic Compounds, U.S.EPA, Office of Solid Waste and Emergency Response, EPA/540/4-91/001, February 1991.

13.4 American Society for Testing and Materials, Method Designation C702, Annual Book of ASTM Standards, Vol. 04.08.2

13.5 American Society for Testing and Materials, Method Designation D6328-98, Annual Book of ASTM Standards, 2001.

13.6 F. F. Pitard, Pierre Gy's Sampling Theory and Sampling Practice, Volume I and II, 1989, CRC Press, Inc., Boca Raton, Florida.

13.7 Methods for the Chemical Analysis of Water and Wastes, US EPA, EPA-600/4-79-020, September 1983.

13.8 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, September 1,1994.

13.9 AML Standard Operating Procedures, Q-Water-RC.

13.10 AML Standard Operating Procedures, Q-WMADISD-RC.

13.11 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, February 1, 2001, Appendix I.

13.12 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Revision 2, June 2002.

STANDARD OPERATING PROCEDURES

No. Q-TemperatureControl-RC

TEMPERATURE MEASUREMENTS AND CONTROL OF OVENS, COOLERS, REFRIGERATORS AND FREEZERS

October 11, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

This Analytical Management Laboratories (AML) standard operating procedure (SOP) is intended to provide guidance for the measurement and tracking of temperature controlled devices such as ovens, refrigerators, freezers, etc. and for the calibration of temperature measurement devices. This SOP is based on ASTM Method E77-98.

2.0 SUMMARY OF METHOD

A number of laboratory operations such as sample digestions are performed at temperatures that are specified by applicable EPA methods. Samples, sample extracts, standards, etc. require storage at controlled temperatures, which require periodic monitoring. A few tests such as the measurement of ignition temperatures require the accurate measurement of temperatures, which are reported as test results. Therefore, glass thermometers used by the laboratory should be checked once per year and dial and electronic thermometers once per quarter, at the temperature used, against an NIST certified thermometer.

NIST certified thermometers do not require additional calibration. Other thermometers may require calibration at the specified intervals. The calibration of such thermometers uses NIST certified thermometers as the reference standard. In addition, ice/water mixtures and/or boiling water/water vapor mixtures are used to verify performance at the temperatures that are commonly used in the laboratory.

3.0 INTERFERENCES

Ice buildup in freezers may result in poor performance of these devices. Periodic defrosting should be performed to restore their efficiency.

4.0 APPARATUS AND MATERIALS

Secondary Standard thermometer (NIST SP250-23 or equivalent) calibrated to 0.5°C or less
Beaker, 250-ml or larger
Laboratory Clamp
Hot Plate

5.0 REAGENTS

ASTM Type II water (ASTM D1193).
Ice: From commercial sources

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Not Applicable.

7.0 HEALTH AND SAFETY

Not Applicable.

8.0 WASTE DISPOSAL

Not Applicable.

9.0 PROCEDURE

9.1 Several types of liquid-in glass thermometers exist. Determine the type of thermometer that is getting calibrated:

9.1.1 Complete-immersion thermometer - These are designed to indicate temperature correctly when the entire thermometer is exposed to the temperature being measured.

9.1.2 Partial-immersion thermometer - These are designed to indicate temperature correctly when the bulb and a specified part of the stem are exposed to the temperature being measured.

9.1.3 Total-immersion thermometer - These are designed to indicate temperature correctly when just the portion of the thermometer containing the liquid (bulb) is exposed to the temperature being measured.

9.1.4 The most common thermometer used in the laboratory is the total-immersion type.

9.2 Visual Inspection of the thermometers - Inspect thermometers for gas bubbles, mercury (or other liquid) separation, glass faults, permanency of pigment in graduations, etc. Cooling the thermometer in a cold bath (Dry ice-acetone or liquid nitrogen) may join separations. Discard thermometers that do not meet visual inspection criteria.

9.3 Thermometer Calibration: Calibrate the thermometers at three temperatures: ice point (0°C), steam point (100°C), and at room temperature (approx. 25°C), when applicable. Check the temperature of the bath with a NIST standard thermometer, verifying the performance of the standard thermometer at the ice point and/or the steam point. Record the serial number of the thermometer to be calibrated and the temperature (Table II) to an accuracy of 0.5°C or less. Successive readings taken one minute apart should agree within on tenth of a division.

9.3.1 For calibration at the ice point, use equal volumes of ice and reagent water in a beaker. Crush ice to small pieces and add only enough water so that the ice does not float. Insert the thermometer, packing the ice gently about the stem, to a depth of approximately one scale division below the ice point (0°C or 32° F). Use a clamp, if necessary. Equilibrate the thermometer(s) for 5-minutes. Record the observed temperature as described above when the readings become stable. NOTE: More than one thermometer may be calibrated at one time using the same bath. It is recommended that the NIST standard thermometer be present at the bath at all times to verify bath performance.

9.3.2 For calibration at the steam point, use approximately 150-ml of Type II water in a 250-ml beaker on a hot plate. Use clamps to support the thermometers so

that the bulbs are immersed in the hot boiling water to a depth of approximately one scale division below the steam point (100°C or 212° F). Equilibrate the thermometer(s) for 5-minutes. Record the observed temperature as described above when the readings become stable. NOTE: More than one thermometer may be calibrated at one time using the same bath. It is recommended that the NIST standard thermometer be present at the bath at all times to verify bath performance. Do not calibrate thermometers used for low temperature measurements (e.g. refrigerators and freezers) at the steam point.

9.3.3 For calibration at the room temperature, immerse the bulbs of the thermometer to be calibrated and a NIST thermometer completely in reagent water at room temperature in a beaker. Equilibrate the thermometer(s) for 5-minutes. Record the observed temperature as described above when the readings become stable. Record the true bath temperature using the NIST standard thermometer.

9.3.4 Electronic thermometers - Use a procedure similar to that described above (Sections 9.3.1 to 9.3.3) for calibrating electronic thermometers.

9.3.5 Frequency of calibration - Calibrate electronic thermometers quarterly. Mercury and alcohol thermometers should be calibrated annually.

9.3.6 Correction Factors: If the measured reading differs by more than 1.0°C from the reference point a correction factor should be applied for measurements made with the thermometer. If the observed temperature is lower (e.g. 1.5°C lower) than the reference point, make a note that reads "Add 1.5°C to the observed temperature". Discard thermometers that differ by more than 2°C.

9.4 Temperature Logs: The temperatures of the sample storage areas are monitored seven days per week using automated continuous monitoring devices. The temperatures of various refrigerators, freezers, ovens and other temperature controlled units should be recorded on a daily basis during weekdays. Record the temperature and the initials on the log (Table I) for each device that requires monitoring both in the morning and in the afternoon. The temperature of the device should be within ±2°C of the stated value by default. Refrigerators should be maintained at 4°C ± 2°C. Freezers should be maintained at -10°C to - 20°C, preferably below -12°C. Make adjustments to the instrument setting, if necessary. Defrost the device if ice buildup is noticed. Notify the laboratory manager if repair or replacement is necessary.

10.0 CALCULATIONS

Record the difference in the readings between the NIST thermometer and the thermometer to be calibrated. If the temperatures differ by more than 1°C, appropriate correction factors should be used.

11.0 QUALITY CONTROL

11.1 The readings of the NIST thermometer at 0°C and/or 100°C are used to measure the accuracy of the calibration procedure.

11.2 Replace the NIST thermometer if the actual temperature readings deviate by more than 0.5°C from the expected value.

11.3 Replace other thermometers if the actual temperature readings deviate by more than 2°C from the expected value.

12.0 REFERENCES

12.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 1, Quality Control, September 1994.

12.2 American Society for Testing (ASTM) Method Designation E77-98.

12.3 AML Standard Operating Procedures, Q-Water-RA.

12.4 EM 200-1-1, Validation of Analytical Chemistry Laboratories, US Army Corps of Engineers. July 1, 1994.

12.5 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, Appendix I, US Army Corps of Engineers, February 1, 2001.

12.6 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Revision 2, June 2002.

Table I
Temperature Monitoring Record (2 pages)

Analytical Management Laboratories
Temperature Record

Device No: _____ Temperature Setting: _____ Range: _____ to _____
Device Description: _____ Thermometer ID: _____ Year: _____

Month:	January			February			March			April			May			June		
	Temp.		Initials	Temp.		Initials	Temp.		Initials	Temp.		Initials	Temp.		Initials	Temp.		Initials
Day	M	E		M	E		M	E		M	E		M	E		M	E	
1																		
2																		
3																		
4																		
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31																		
Supervisor Review																		

NOTE: Record temperatures to an accuracy of ±0.5°C.

Analytical Management Laboratories
Temperature Record

Device No: _____ Temperature Setting: _____ Range: _____ to _____
Device Description: _____ Thermometer ID: _____ Year: _____

Month:	July			August			September			October			November			December		
	Temp.		Initials	Temp.		Initials	Temp.		Initials	Temp.		Initials	Temp.		Initials	Temp.		Initials
Day	M	E		M	E		M	E		M	E		M	E		M	E	
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Supervisor Review																		

NOTE: Record temperatures to an accuracy of ±0.5°C.

Table II
Temperature Calibration Log (1 page)

Temperature Calibration Log
Analytical Management Laboratories

Date: _____ Serial No. of NIST Thermometer: _____

Thermometer ID (e.g. Serial No.)	Type of Bath & Reference Temp.	Actual temperature Reading (°C)	Difference (Actual-Ref.)	Correction to be Applied
NIST Reference	Ice Point (0°C)			NA
NIST Reference	Steam Point (100°C)			NA
NIST Reference	Room Temp. (___°C)		NA	NA

NOTE: Record temperatures to an accuracy of ±0.5°C.

Analytical Management Laboratories

Training Log

Date: _____

Provided by: _____

Topic: _____

ATTENDEE NAME	ATTENDEE SIGNATURE

STANDARD OPERATING PROCEDURES

No. Q-Training-RF

TECHNICAL TRAINING

October 11, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

TECHNICAL TRAINING

1.0 Scope and Application

Analyses and associated quality-related activities which are performed by Analytical Management Laboratories (AML) should be conducted by personnel who have been trained and qualified based on education, experience, external and/or internal training, and evaluation by appropriate processes. Training and qualifications should be documented and maintained as described in this AML standard operating procedure (SOP). This SOP establishes the minimum requirements for technical training and associated documentation.

2.0 Method Summary and Definitions

2.1 The laboratory develops and maintains capabilities in various test areas using acceptable procedures. New laboratory personnel are trained by experienced personnel. New employees are then qualified by the Team Leader after training has been completed. Existing employees maintain their qualifications through periodic demonstration of their capabilities. Training (i.e., on-the-job, external and continuing education) should be documented in individual employee training files.

2.1.1 Laboratory Capabilities: The demonstrated capability of the laboratory to perform a test in accordance with acceptable standards, which may involve initial demonstration of capabilities, and continuing demonstration through participation in external PT sample studies, etc. The laboratory capability to perform a given test may be derived from the capabilities of one or more "Work Cells" or one or more individual analysts.

2.1.2 Work Cell Capabilities: A Work Cell is defined as a group of analysts and/or technicians with specifically defined tasks that together perform a test method. In large laboratories, several work cells may perform the same test. In small laboratories, one work cell may perform several tests. Personnel in a work cell may perform specialized tasks that may consist of sample preparation only, instrument operation only, data processing and report preparation only, etc.

2.1.3 Analyst Capabilities: The capability of an analyst to perform all the functions of a test, or assigned functions if part of a work cell.

3.0 Health and Safety

Not Applicable.

4.0 Sample Preservation, Containers, Handling, and Storage

Not Applicable.

5.0 Interferences and Potential Problems

If procedures are performed by unqualified personnel, data quality may be adversely affected.

6.0 Equipment/Apparatus

Not Applicable.

7.0 Reagents

Not Applicable.

8.0 Procedure

Technical training at AML may be performed as orientation, on a personal basis (one on one), or through participation in classes/presentations. Technical training includes QA training, health and safety training, external training, and analyst qualification.

8.1 **Quality Assurance Training**.

AML has instituted a program that covers the general principles of regulatory requirements, basic quality control procedures, responsibilities of the laboratory staff, the reporting of nonconformances, principles and applications of audits, QC samples, control charts, laboratory documentation, data integrity, and general ethics. The following are addressed:

8.1.1 New employees should be instructed to read the AML Quality Assurance Plan (QAP), to be completed within 30 days of the start work date.

8.1.2 Each new employee should receive an orientation by the QA Manager within 30 days of the start work date. This orientation should discuss the general aspects of the laboratory QA program listed above, good laboratory practices, and operational SOPs.

8.1.3 The QA Manager should sign off on the Employee Training Summary form (see Figure 1) indicating that the information has been read and understood (e.g., QAP) and that the orientation has been performed.

8.1.4 The list of QC/QA and analytical procedure specific operational SOPs shown in Appendices 1 to 5 should be reviewed in order to advise the new employee on laboratory procedures that are applicable in the performance of duties.

8.2 **Health and Safety Training**

AML has implemented a program that covers training and qualifying a new employee on having sufficient knowledge of the laboratory's health and safety practices. The program addresses the following:

8.2.1 New employees should be instructed to read the laboratory's Chemical Hygiene Plan and Waste Disposal Plan within 30 days of the start of work date.

8.2.2 The Health and Safety Officer should give orientation describing the laboratory's safety aspects within 30 days of the start work date.

8.2.3 The Health and Safety Officer should sign off on the Employer Training Summary (see Figure 1) indicating that all the information has been read and understood (e.g., Chemical Hygiene Plan, Waste Disposal Plan) and that the orientation has been performed.

8.3 External Training

Training received outside the laboratory should be documented. This includes instrument manufacturer's courses, as well as professional development training (e.g., management training, effective speaking training, etc.). Acceptable forms of documentation include the following: certificates, instructor letters, course registration papers, diplomas, awards, etc.

8.4 Analyst Qualification

For each method/SOP performed, the laboratory should generate and maintain documentation that an analyst is qualified to perform that method/SOP within the precision and bias limits specified by the laboratory (control charts) or in the method. The analyst qualification may include performing all the functions of test, unless a work cell (see Section 2.1.2) is employed in the performance of the test. The performance of the work cell should be documented and linked to the training records of the individual members of the work cell. The following procedures should be used to perform analyst/work cell qualifications.

8.4.1 The supervisor should establish Trainer(s) for each procedure in which an analyst requires training.

8.4.2 The analyst (Trainee) should read the applicable method and/or SOP (See Appendices 2 to 5 for a list of SOPs) and demonstrate an understanding of the material.

8.4.3 The analyst should perform the method or SOP under the direct supervision of the Trainer, a senior analyst, and/or the Team Leader using a minimum of 4 aliquots of a laboratory control sample (QC reference sample) containing each analyte of interest (refer to specific method for analytes).

8.4.3.1 These samples are to be analyzed by the same procedures that will be used to prepare and analyze actual samples. Each method should be evaluated using the same matrix as the samples to which the method will be applied (water or soil). Reagent water, clean sand or other control matrices should be used. The concentration to be spiked is dependent on the specific procedures to be used. Refer to the specific method/SOP for guidance.

8.4.3.2 All batch QC requirements should be performed and they should meet method/SOP requirements.

8.4.3.3 The Trainer should calculate the average recovery (x) and the standard deviation (s) of the recoveries for each analyte of interest using the four results.

8.4.3.4 For each analyte, the Trainer should compare s and x with the corresponding acceptance criteria for precision and accuracy, as determined from laboratory control charts, if available, or those

criteria found in each of the determinative methods. If s and x for all analytes of interest meet the acceptance criteria, the analyst performance is acceptable and the analyst is deemed qualified to perform the method/SOP. If any individual s exceeds the precision limit or any individual x falls outside the range of accuracy, then the analyst performance is unacceptable for that analyte and additional training should be provided to the analyst until satisfactory performance is attained.

8.4.4 In some instances, an analyst may be deemed qualified for a particular method based on prior experience and a demonstrated understanding of the procedure. In other instances, method/SOP review or supervisor review during performance may substitute for the initial demonstration of capability by the analyst. The appropriate Team Leader and/or the AML Operations Manager are responsible for determining the method of qualification.

8.4.5 The analyst should continue to demonstrate capability using one or more of the following methods: (1) by successfully analyzing external PT samples (e.g. WS, WP, and RCRA solids) annually with semi-annual frequency preferred; (2) by successfully analyzing laboratory control samples on a monthly basis; and (3) by maintaining the laboratory capability as described in Section 8.4.6.

8.4.6 Each test area, the responsible analyst should demonstrate the continuing capability of the analyst and the laboratory to perform the test method by ensuring that applicable SOPs are kept current and represent the laboratory's current implementation of the method. The sensitivity of each method should be demonstrated on a quarterly basis by analyzing a RL check sample or the MDL check sample as applicable. The precision/bias of the method should be demonstrated by analyzing laboratory control samples with each batch of samples processed, and external PT samples or other QC check samples at the applicable frequency.

8.4.7 The analysts should complete documentation that demonstrates that each analyst has read, understood, and is using the latest version of the laboratory’s in-house documentation that includes method SOPs, laboratory quality assurance manual and other applicable quality documentation.

8.5 Laboratory Information Management Systems (LIMS) Training

AML has a LIMS Manager with extensive background, experience and/or training in environmental laboratory analysis, sample management, and laboratory management to perform data management and reporting functions described in the laboratory QAP. The LIMS Manager should provide additional training to analysts in the use of the LIMS. This LIMS training should supplement training provided by the technical Trainer(s) in the use of LIMS to record and report data generated by AML.

8.6 Computer Training

The analyst may be required to use Microsoft® (MS) Office™ products in the normal performance of the duties. This may include MS Word, Excel, Access, Outlook, etc. Excel, the MS Spreadsheet application contains a number of statistical functions that are commonly used to perform calculations that are described in Section 9. The LIMS Manager and/or the Team Leader should provide this training to new analysts. Training may include external training for new analysts that are not familiar with computer use. Commonly used functions are shown below:

8.6.1 ***Single Array Functions.*** These functions are commonly used in the determination of instrument detection limits (IDL), method detection limits (MDL), etc., and in control charting. Commonly used functions are shown below:

8.6.1.1 **AVERAGE**(number 1, number 2, etc.). This function returns the average (arithmetic mean) of its arguments, which may be numbers or references to an array that contain the numbers. It ignores logical values and text that may be included in the array.

8.6.1.2 **STDEV**(number 1, number 2, etc.). This function returns the standard deviation of its arguments, which may be numbers or references to an array that contain the numbers. It ignores logical values and text that may be included in the array.

8.6.1.3 **VAR**(number 1, number 2, etc.). This function returns the variance (square of the standard deviation) of its arguments, which may be numbers or references to an array that contain the numbers. It ignores logical values and text that may be included in the array.

8.6.1.4 **SUM**(number 1, number 2, etc.). This function returns the sum of its arguments, which may be numbers or references to an array that contain the numbers. It ignores logical values and text that may be included in the array.

8.6.1.5 **COUNTA**(value1, value 2, etc.). This function returns the number of cells that are not empty. Arguments may be numbers or references to an array that contain the numbers or values.

8.6.1.6 **MAX**(number 1, number 2, etc.). This function returns the largest value among the arguments, which may be numbers or references to an array that contain the numbers. It ignores logical values and text that may be included in the array.

8.6.1.7 **MIN**(number 1, number 2, etc.). This function returns the smallest value among the arguments, which may be numbers or references to an array that contain the numbers. It ignores logical values and text that may be included in the array.

8.6.2 ***Functions Requiring Two Arrays.*** These functions are commonly used in instrument calibration. They require an array containing known y-values and an array containing known x-values. Commonly used functions are shown below:

8.6.2.1 **SLOPE**(known_y's, known_x's). This function returns the slope (m)

of the least squares best fit linear regression line (y = m*x + c) from a list of arguments for y and x or references to the two arrays designated as y and x that contain the numbers.

8.6.2.2 **INTERCEPT**(known_y's, known_x's). This function returns the y-intercept (c) of the least squares best fit linear regression line (y = m*x + c) from a list of arguments for y and x or references to the two arrays designated as y and x that contain the numbers.

8.6.2.3 **CORREL**(known_y's, known_x's). This function returns the correlation coefficient (c) of the least squares best fit linear regression line (y = m*x + c) from a list of arguments for y and x or references to the two arrays designated as y and x that contain the numbers. Correlation coefficients are independent of the array designation for x and y.

8.6.2.4 **FORECAST**(x**,** known_y's, known_x's). This function returns the y value from the least squares best fit linear regression line (y = m*x + c) from a list of arguments for y and x or references to the two arrays designated as y and x that contain the numbers. For example, if the observed absorbances (x-axis) for a series of standards with known concentrations (y-axis) are determined, the concentration (y) of an unknown can be calculated from the measured absorbance (x). The equation for linear regression is used to perform calculations.

8.7 Technical Training Documentation

Training activities should be documented and maintained in the Individual's Training File maintained by the Human Resources Manager. These files are divided into the following categories.

8.7.1 Resume, academic credentials, employment history, professional memberships/registrations.

8.7.2 Employee Training Summary. Document general employee orientation, QC/QA and Health and Safety orientation, and ethics agreement, using the form in Figure 1. This form is also used to summarize internal and external training, as well as those procedures for which the employee has been deemed qualified.

8.7.3 Analyst Qualification is performed and documented using the forms shown in Figures 2 and 3. Figure 2 documents the actual training, while Figure 3 documents employee's acceptance of the AML SOPs to guide employee's performance in technical areas.

8.7.4 External and Internal Training. Documentation may include certificates, instructor letters, diplomas, awards, group training attendance records, individual training attendance records (see Figures 4 and 5, respectively).

8.7.5 Each employee should review their training file annually for accuracy and completeness.

9.0 Calculations

Common calculations done by the analysts in the normal performance of duties are shown in this section. The analysts should refer to specific method/SOP for details that are not included in this section.

9.1 The mean (x) of the results is calculated as follows:

$$x = \frac{x_1 + x_2 + x_3 + x_n}{n}$$

where:

x_1, x_2, x_3, x_n = individual results, and
n = the number of results

$$s = \sqrt{\sum_{i=1}^{n} \frac{(x_i - x)^2}{n-1}}$$

9.2 The standard deviation(std dev, S.D. or s) of the results is calculated as follows:

where:

s = standard deviation
x_i = measured value of the *i*th replicate
x = mean of replicate
n = number of replicates

9.3 Precision for environmental analysis is often expressed as the relative percent difference (RPD) for a set of duplicate measurements of a variable (X). RPD is defined as follows:

$$RPD = \frac{|x_1 - x_2|}{(x_1 + x_2)/2} x\,100$$

9.4 Precision may also be expressed as the standard deviation (SD) as described above, percent relative standard deviation (RSD), or coefficient of variation (CV).

$$RSD = CV = \frac{[std\ dev]}{\bar{x}} x\,100$$

9.5 Percent recovery (%R) for spiked samples (e.g. matrix spikes) is calculated as follows.

$$\%R = \frac{(x_s - x_u)}{K} x\,100$$

where:

X_s = measured value of the spiked sample

X_u = measured value of the unspiked sample

K = Amount spiked to the sample

When calculating %R for laboratory control samples (LCS), blank spikes or other reference materials, X_u could be set at zero.

9.6 The relationship between percent bias (%B) and percent recovery is as follows:

$$\%B = |\%R - 100|$$

9.7 Calculation of calibration factors (CF):

Calibration factors are normally used in organic analysis for external standard calibration method. They are calculated by tabulating the peak area against concentration for each analyte in each standard for multi-concentration initial calibration (ICAL). Calculate calibration factors (CF) for each analyte as shown below:

$$CF = (A_x) / (C_x)$$

where:

Ax = Peak area for the analyte being measured
Cx = Analyte concentration (e.g. ng/mL, mg/L, etc.) in the standard

9.8 Calculation of mean calibration factors (mean CF or CF_m)

The mean or average calibration factor (mean CF = CF_m) is calculated as shown below:

$$\text{mean CF} = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

Where CF_i are the calibration factors for each ICAL standard.

9.9 Calculation of %D for calibration verification

The mean calibration factor from the ICAL is normally used in calculating sample concentrations. The validity of the ICAL is determined by analyzing calibration verification solutions (CALV) with sample analysis. The percent difference (%D) between the observed calibration factor for CALV (CF_v) and the mean calibration factor from the ICAL (CF_m) are calculated as shown below:

$$\%D = ((CF_v - CF_m) * 100 / (CF_m))$$

9.10 Calculation of %Drift for calibration verification

Calculation of %Drift is an alternate procedure for calibration verification. This procedure is primarily used when linear regression (common for inorganic analytical procedures) is used for ICAL instead of the CF or RF methods for sample quantitattion. The percent drift is calculated from the difference between the measured concentration for CALV (C_v) and its true concentration as shown below:

$$\% \text{ Drift} = ((C_v - C_m) * 100) / (C_m)$$

where:
C_m = True concentration of the CALV standard.
C_v = Measured concentration of the CALV standard using selected quantitation method (e.g. linear regression).

9.11 Calculation of sample concentrations in water samples when calibration factors (CF) are used. For water samples the calculation is as follows:

$$\text{Concentration} = \frac{(Ax * V_e * D)}{(CF_m * V_s)}$$

where:
A_X = Peak area of the analyte being measured.
CF_m = Mean calibration factor for the analyte being measured
V_e = Volume of the final extract
V_s = Volume of water extracted
D = Dilution factor for the analysis

9.12 Calculation of sample concentrations in solid (soil, sediment or waste) samples when calibration factors are used. For solid samples the calculation is as follows:

$$\text{Concentration} = \frac{(Ax * V_e * D * 100)}{(CF_m * W_s * PS)}$$

where:
A_X = Peak area of the analyte being measured.
CF_m = Mean calibration factor for analyte being measured
V_e = Volume of the final extract
W_s = Weight of solid sample extracted
D = Dilution factor for the analysis
PS = Percent solids

9.13 The response factors (RF) are normally used in organic analysis for internal standard calibration method. They are calculated by tabulating the peak area

against concentration for each analyte in each standard for multi-concentration initial calibration (ICAL). Calculate response factors (CF) for each analyte as shown below:

$$RF = (A_x * C_{is}) / (A_{is} * C_x)$$

where:
Ax = Area of the chromatographic peak (characteristic ion chromatogram for GC/MS methods) for the analyte being measured
Ais = Area of the chromatographic peak (characteristic ion chromatogram for GC/MS methods) for the associated internal standard
Cis = Concentration of the internal standard
Cx = Concentration of the analyte being measured

9.14 Calculation of sample concentrations in water samples when response factors (RF) are used. For water samples the calculation is as follows:

$$\text{Concentration} = \frac{(A_X) * (C_{IS}) * Ve * D}{(A_{IS})\,(RF_m)\,(V_o)}$$

where:

Ax = Area of the chromatographic peak (characteristic ion chromatogram for GC/MS methods) for the analyte being measured in the sample
Ais = Area of the chromatographic peak (characteristic ion chromatogram for GC/MS methods) for the associated internal standard in the sample
Cis = Concentration of the internal standard in the sample extract
RF_m = Mean response factor from the ICAL for the analyte being measured
V_e = Volume of the sample extract
V_o = Initial volume of the water sample that was extracted
D = Dilution factor, if any

9.15 Calculation of sample concentrations in soil samples when response factors (RF) are used. For soil samples the calculation is as follows:

$$\text{Concentration} = \frac{(A_X) * (C_{IS}) * Ve * 100 * D}{(A_{IS})\,(RF_m)\,(W_s) * PS}$$

where:

Ax = Area of the chromatographic peak (characteristic ion chromatogram for GC/MS methods) for the analyte being measured in the sample
Ais = Area of the chromatographic peak (characteristic ion chromatogram for GC/MS methods) for the associated internal standard in the sample
Cis = Concentration of the internal standard in the sample extract
RF_m = Mean response factor from the ICAL for the analyte being measured

V_e = Volume of the sample extract
W_s = Initial weight of the soil sample that was extracted
D = Dilution factor, if any
PS = percent solids

9.16 Calculation of sample concentrations from linear regression.
Least squares procedures to best fit x (e.g. concentrations) and y (e.g. instrument response) arrays for linear regression are too complicated to describe in this section. These procedures should be performed either using a scientific electronic or preferably using an Excel spreadsheet as described in Section 8.6.2. Spreadsheets may be used for calculating the slope, y-intercept and the correlation coefficient for the linear regression line and for calculating (or forecasting) sample concentrations as described in Section 8.6.2. Spreadsheets may also be used for graphing the ICAL and to show the trend line.

10.0 Quality Assurance/Quality Control

10.1 Each employee must review their Individual Training File annually for accuracy and completeness.

10.2 All training and qualification files shall be kept in a readily accessible location while the employee is employed with AML. Upon termination of employment, the records should be kept indefinitely, but may be placed in archived file locations.

11.0 Data Validation

Not Applicable

12.0 Waste Disposal

Not Applicable

13.0 References

13.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Update IV, Chapter 1.
13.2 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000B, Determinative Chromatographic Separations, December 1996.
13.3 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

13.4 AML Standard Operating Procedures, Q-MDL-LLR-RL-MQL-RB.
13.5 AML Standard Operating Procedures, Q-ControlCharts-RC.
13.6 AML Standard Operating Procedures, Q-SOPs-RD.
13.7 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.
13.8 EM 200-1-2, Technical Project Planning (TPP) Process, US Army Corps of Engineers, August 31, 1998.
13.9 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, US Army Corps of Engineers, September 1,1994. Appendix I.
13.10 EM 200-1-10, Guidance for Evaluating Performance-Based Chemical Data, Department of the Army, 28 December 2001.

Analytical Management Laboratories
Employee Orientation and Training Summary

Employee Name: _____

Laboratory Section: _____

Start Date: _____

Supervisor: _____

New Employee Orientation

Training Topic	Date	Trainer
AML General		
AML Employee Benefits (IRS/INS Forms)		
AML Ethics Policy		
AML Customer Service Philosophy		
AML QC/QA Orientation		
AML Health & Safety Orientation		
AML LIMS Orientation		
AML Computer Orientation		

Internal Technical Training

Training Topic or SOP Number	Period	Trainer

External Technical Training

Training Topic	Date	Related Info. (describe)

Figure 1. Employee Training Summary

Analytical Management Laboratories
Employee Training Log

Name of Trainee:		
Name of Trainer:		
Analytical Procedure:		
Matrix:		
This is to certify that the trainee has successfully completed the following steps in the training:		
1. Has reviewed the method and SOP.	Initials:	Date:
2. Has been shown all steps in the test.	Initials:	Date:
3. Has performed the test under supervision.	Initials:	Date:
4. Can independently perform the test.	Initials:	Date:
5. Has successfully analyzed spike/PT samples. The analyst's results, true values and acceptance limits are shown in the attachment.	Initials:	Date:

Date	Trainer's Comments	Proposed Corrective Action

This is to certify that the trainee can perform the above method without supervision.

Date	Signature
	Trainee:
	Trainer:
	QA Officer:

Figure 2. Employee Training Log

Analytical Management Laboratories
SOP Implementation Log

Name of Analyst:
Name of Supervisor:
SOP Number:
SOP Title:
Referenced Method #1:
Referenced Method #2:
Referenced Method #3:
Referenced Method #4:
Referenced Method #5:
This is to acknowledge that I have reviewed the Analytical Management Laboratories (AML) Standard Operating Procedure (SOP) described above and the associated EPA methods (SW-846, CWA, SDWA methods, Standard Methods, etc.) pertaining to this SOP. I agree to access the SOP and/or the referenced methods from their current locations (personal copy, AML bookshelves, internet, etc.). I agree to implement the SOP at AML in accordance with project specific requirements. I understand that required documentation may be more or less than those stated in the AML SOP and I agree to follow directions provided by my supervisor or his designee (e.g. Project Manager) for specific projects.

Date	Signature
	Analyst:
	Supervisor:
	QA Manager:

Figure 3. SOP Implementation Log

Analytical Management Laboratories

Group Training Attendance Record

Subject:	
Speaker(s):	Date:
Name of the Attendee (Last name, first name)	**Signature**

Figure 4. Group Training Attendance Record

Analytical Management Laboratories
Individual Training Attendance Record

Employee: _____

Date of Training: _____

Subject of Training: _____

Type of Training:

_____On-the-Job

_____Electronic

_____Video Tape

_____Audio Tape

_____Computer

_____One on one

Training Administered by:

Figure 5. Individual Training Attendance Record

Appendix 1

List of AML QC/QA SOPs

List Of AML QC/QA SOPs

SOP Number	SOP Rev.	SOP Title
Q-Audit	RD	Internal Quality Assurance (QA) Audits and Performance Evaluation
Q-Cleaning	RC	Glassware Cleaning Procedures
Q-Control Charts	RC	Generation and Maintenance of Control Charts
Q-Complaint	RB	Complaint Resolution
Q-DataMgmt	RD	Data and Information Storage, Retrieval, and Management Procedures
Q-Data Verification	RB	Data Reduction, Review, Verification, and Reporting Procedures
Q-EDD	RC	Electronic Data Deliverables
Q-Ethics	RC	Ethics Statement
Q-logbooks	RB	Lab Notebooks Control, Use, Review, and Approval
Q-SampleMgmt	RB	Sample Receiving, Log-in, Custody, and Tracking
Q-Manual Integration	RB	Manual Integration
Q-MDL-LLR-RL-MQL	RB	IDL, MDL, LLR, RL, PQL, and Initial Demonstration of Performance
Q-ProjectMgmt	RC	Project Management
Q-Rounding	RA	Significant Figures
Q-Software	RC	Software Training, Use, and Evaluation
Q-Sonicator	RD	Sonicator Tuning
Q-SOPs	RD	Preparation, Distribution, and Tracking of Standard Operating Procedure (SOPs) Documents
Q-Standards	RB	Analytical Standards and Reagents - Preparation, Labeling, Monitoring, Traceability, and Storage
Q-Subsampling	RC	Subsampling of Containers
Q-Temperature Control	RB	Temperature Measurements
Q-Training	RE	Technical Training
Q-Volumes	RB	Volumetric Measurements
Q-Water	RC	Reagent Water
Q-Gravimetric	RB	Balances and Gravimetric Measurements
Q-WasteMgmt	RB	Waste Management and Disposal Including Sample Disposal

Appendix 2

List of AML Organic Analysis SOPs

List of AML Organic Analysis SOPs

SOP Number	SOP Rev.	SOP Title
O-5035	RA	Soil sample collection and preservation procedures for the analysis of VOCs by SW-846 Method 5035
O-8260	RE	Volatile Organic Compounds by GC/MS capillary column method
O-8270	RE	Semivolatile organic compounds by GC/MS capillary column procedure
O-8081	RC	Organochlorine pesticides by GC/ECD
O-8082	RD	Polychlorinated biphenyls (PCBs) by GC/ECD
O-8151	RB	Chlorophenoxy herbicides by GC/ECD
O-8310	RB	Polynuclear aromatic hydrocarbons by HPLC
O-8330	RC	Niroaromatics, nitramines, and related explosive compounds by HPLC
O-TPHD	RB	Total petroleum hydrocarbons (TPH) Diesel fraction by GC/FID: capillary column technique
O-TPHG	RE	Total petroleum hydrocarbons (TPH) Gasoline fraction by GC/FID: capillary column technique
O-MEE	RA	Methane, ethane, ethene and related natural gas analysis by GC/FID
O-Hydrogen	RA	Hydrogen Analysis by GC/TCD

Appendix 3

List of AML Inorganic Analysis SOPs

List of AML Inorganic Analysis SOPs

SOP Number	SOP Rev.	SOP Title
I-6010	RB	Metals analysis by inductively coupled plasma - atomic emission spectroscopy (ICP/AES)
I-6020	RB	Metals analysis by inductively coupled plasma - mass spectrometry (ICP/MS)
I-7000	RB	Metals analysis by graphite furnace atomic absorption spectroscopy (GFAA)
I-7470-7471	RB	Mercury in soil and water by cold vapor atomic absorption (CVAA)
I-U-Th-6020	RA	Analysis for thorium and uranium isotopes by inductively coupled plasma mass spectrometry (ICP/MS)
I-3010	RC	Acid digestion of aqueous samples for total metals analysis by flame atomic absorption spectroscopy (FLAA) or ICP/AES
I-3020	RD	Acid digestion of aqueous samples for total metals analysis by GFAA or ICP/MS
I-3050	RC	Acid digestion of soil, sediment, and sludge samples for total recoverable metals analysis by FLAA, GFAA, ICP/AES or ICP/MS
I-9056	RD	Determination of inorganic anions by ion chromatography (IC)
I-9010-9014	RA	Determination of total or amenable cyanide by distillation and manual spectrophotometric analysis
I-9060	RA	Total Organic carbon
I-7196	RA	Hexavalent chromium analysis in aqueous samples by manual spectrophotometry
I-3060-7196	RA	Hexavalent chromium analysis in soil, sediment, and waste samples by alkaline digestion and manual spectrophotometry
I-NC	RA	Nitrocellulose analysis by spectrophotometry

Appendix 4

List of AML Waste Testing SOPs

List Of AML Waste Testing SOPs

SOP Number	SOP Rev.	SOP Title
W-1010-1020	RA	Ignitability
W-9040-045	RA	Soil, water, and waste pH
W-9010-9030	RA	Reactivity - cyanide and sulfide
W-9081	RA	Cation exchange capacity (CEC) of soils by sodium acetate treatment and ICP/AES analysis
W-9095	RA	Paint filter liquids test
W-1311	RA	Toxicity chracteristic leaching procedure (TCLP)
W-1312	RA	Synthetic precipitation leaching procedure (SPLP)

Appendix 5

List of AML Wastewater SOPs

List Of AML Wastewater SOPs

SOP Number	SOP Rev.	SOP Title
WW-110-3	RA	Color by spectrophotometry
WW-120-1	RA	Specific conductance
WW-130-1	RA	Hardness, total, by ICP/AES and calculations
WW-150-1	RA	Electrometric pH measurements
WW-160-1	RA	Residue, filterable, gravimetric or total dissolved solids (TDS)
WW-160-2	RA	Residue, non-filterable, gravimetric or total suspended solids (TSS)
WW-160-3	RA	Residual, total, gravimetric or total (TS)
WW-160-4	RA	Residue, volatile, gravimetric or total volatile solids (TVS)
WW-160-5	RA	Residue, settleable matter, volumetric, Imhoff cone
WW-170-1	RA	Temperature
WW-180-1	RA	Turbidity
WW-305-1	RA	Acidity, titrimetric
WW-310-1	RA	Alkalinity, titrimetric
WW-325-3	RA	Chloride by titrimetry
WW-330-5	RA	Chlorine, total residual by spectrophotometry
WW-335-1	RA	Cyanide, amenable to chlorination by spectrophotometry
WW-335-2	RA	Cyanide, total by spectrophotometry
WW-350-2	RA	Ammonia, colorimetric, distillation procedure
WW-351-3	RA	Total Kjeldahl nitrogen (TKN) by block digestion, colorimetric
WW-353-3	RA	Nitrate and nitrite, manual cadmium reduction, colorimetric
WW-354-1	RA	Nitrite, colorimetric
WW-360-1	RA	Oxygen, dissolved by membrane electrode
WW-365-2	RA	Phosphorus, total, block digestion, colorimetric
WW-375-4	RA	Sulfate, turbidimetric
WW-376-2	RA	Sulfide, colorimetric

List Of AML Wastewater SOPs (contd.)

SOP Number	SOP Rev.	SOP Title
WW-405-1	RA	Biochemical oxygen demand (BOD)
WW-410-4	RA	Chemical oxygen demand (COD), colorimetric, manual
WW-1664	RA	Oil and grease, gravimetric
WW-415-1	RA	Organic carbon, total
WW-420-1	RA	Phenolics, total recoverable, colorimetric, manual distillation

STANDARD OPERATING PROCEDURES

No. Q-Volumetric-RC

VOLUMETRIC MEASUREMENTS AND CONTROL OF VOLUME MEASUREMENT DEVICES

October 31, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

1.1 This Analytical Management Laboratories (AML) standard operating procedure (SOP) describes procedures for the calibration and use of volume measurement devices such as pipettes, syringes, burettes, measuring cylinders, volumetric flasks, etc. that are routinely used in laboratory analysis.

1.2 This SOP is based on USACE and EPA guidance documents and ASTM methods E542, E969, E694, E1154, E1293, E1272, E1878, E288, and D5522-99a.

1.3 Professional judgment should be used in applying guidance contained in this SOP for calibrating apparatus with capacities below 0.1-ml.

2.0 SUMMARY OF METHOD

2.1 Volumetric measurements are important components in generating accurate analytical data. Accurate volume measurements are needed for a number of operations such as the measurement of initial sample volumes, final sample volumes, delivery of various amounts of spiking solutions, and in the preparation of standard solutions.

2.2 Although some volume measurements may be made without recalibration of some of the measurement devices, the volume markings cannot be taken at face value for all devices and for all applications.

2.3 Calibration of measurement devices are based on accurate weight measurements on volumes delivered by and/or contained in the volume measurement device using water as the reference material. Then, with the density of water known, the correct volume is found. The effect of other parameters such as relative humidity, air temperature, air pressure, etc. on volumetric tolerance is negligible compared to the water temperature.

2.4 The unit, milliliter (mL) is considered as equivalent to the cubic centimeter (cm^3), which is the most commonly used SI unit for volume. The basic SI unit for volume is the cubic meter (m^3).

3.0 INTERFERENCES

3.1 A number of extraneous conditions can influence the accuracy and precision of volume measurement devices. They include the following:

- 3.1.1 The type of device - to contain (TC) or to deliver (TD)
- 3.1.2 Cleanliness of the internal glassware surface of the device
- 3.1.3 The nature, color and other physical properties (e.g. viscosity) of the fluid
- 3.1.4 Temperature
- 3.1.5 Method of reading
- 3.1.6 Type of meniscus
- 3.1.7 Grade or Class (e.g. Class A) of glassware/device.

3.2 The coefficient of expansion of glass is sufficiently small that the correction (1part per 10,000 for a change of 5°C) required for using devices calibrated at 20°C at temperatures (e.g. ambient laboratory temperature) other than 20C is negligible.

3.3 Proper selection of the measurement device for the intended operation and adequate training of personnel are necessary to minimize and/or eliminate problems involving imprecise volume measurements.

4.0 APPARATUS AND MATERIALS

4.1 Analytical Balance (to 0.1-mg accuracy)
4.2 Devices and apparatus requiring calibration

5.0 REAGENTS

5.1 ASTM Type II water (ASTM D1193)
5.2 ASTM Type II water (See AML SOP on reagent water)

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Not applicable

7.0 HEALTH AND SAFETY

Not applicable

8.0 WASTE DISPOSAL

Not applicable

9.0 PROCEDURE

9.1 Classes of Volumetrics: Volumetrics come in two classes, Class-A and Class-B. Class-A volumetric devices have stringent specifications for tolerance in addition to requirements on type of glass, graduation spacing, graduation pattern, etc. Class-B volumetrics are general purpose instruments with the same basic design with a tolerance range twice that of Class-A devices. See figure in Appendix 1 for an example (reproduced from ASTM Method E694) of inscriptions on volumetric apparatus.

9.2 Types of Volumetrics: Volumetric devices may be made to contain (TC) or to deliver (TD). Volumetrics that are made to contain include volumetric flasks, beakers, Erlenmeyers, measuring cylinders, etc. Volumetrics that are made to deliver include transfer pipettes, burettes, Eppendorf or piston/plunger operated volumetric apparatus (POVA). For this SOP, the volumetrics are divided into two groups, standard and nonstandard:

9.2.1 Standard Volumetrics: Volumes measured using these devices are often included in calculations. These include disposable and reusable pipettes, burettes, measuring cylinders, and fixed and variable volume Eppendorf-type (POVA) pipettes. The USACE recommended tolerance for standard volumetrics is <±1%.

9.2.2 Nonstandard Volumetrics: Volumes measured using these devices may not always be included in calculations with a few exceptions. These include K-D tubes, Zymark tubes, plastic cups, centrifuge tubes, culture tubes, etc. The USACE recommended tolerance for nonstandard volumetrics is <±3%.

9.3 Tolerance Specifications: ASTM has tolerance specifications for the performance of volumetric devices. These are included in Appendix 2. See referenced ASTM method for more details. These tolerance specifications are strictly applicable to the manufacturer of these devices. The AML tolerance specifications for normal use of these devices are described for standard (<±1%) and nonstandard (<±3%) volumetrics are Sections 9.2.1 and 9.2.2 respectively.

9.4 Initial Inspection: All volumetric devices, glassware, or labware should be initially inspected, and all cracked or damaged items removed from use. Dirty items should be returned to the washing station to be cleaned in accordance with the referenced AML SOP on this topic.

9.5 Recalibration or Calibration Verification Policy:

9.5.1 Class A devices: The calibration of "Class A" commercial glassware marked with a large "A", which should meet U.S. Federal specifications can be accepted, and the glassware can be used without recalibration. This includes most volumetric flasks, which have been calibrated to contain (TC) fixed volumes (100-mL, etc.) and certain pipettes that have been calibrated to deliver (TD) fixed volumes (5-mL, etc.) at 20°C. The tolerance values established by the National Bureau of Standards (NBS) are shown on Table I. The calibration of Class devices should be checked when received prior to use at AML. Check one device for each lot or package of devices. Document calibration checks as shown in Table III.

9.5.2 Glassware that are not Class A will require verification and/or recalibration depending on the intended application. The calibration of non-Class A devices should be checked when received prior to use at AML. Check one device for each lot or package of devices. Document calibration checks as shown in Table IV. See Section 9.5.3 for continuing verification.

9.5.3 Calibration Frequency: Use USACE guidance for frequency of calibration. Repeat calibration verification more frequently, if necessary.

9.5.3.1 Variable volume Eppendorf-type pipettes - Weekly
9.5.3.2 Syringes - Monthly
9.5.3.3 Fixed volume Eppendorf-type pipettes - Monthly
9.5.3.4 Fixed volume autopipettors, diluters, and dispensers - when pressed into service and annual thereafter
9.5.3.5 Graduated cylinders used for sample volume measurements - when pressed into service and annual thereafter
9.5.3.6 Environmental Express plastic cups - once per batch upon receipt
9.5.3.7 Disposable transfer pipettes - once per batch upon receipt
9.5.3.8 Class A pipettes and burettes, etc. - when pressed into service
9.5.3.9 Beakers and Erlenmeyers - as needed

9.5 **Cleanliness:** Devices that do not drain uniformly, leaving droplets of water adhering to the inner surface require cleaning. Cleaning can be done with a warm detergent solution, other

cleaning agents, solvents, etc. The devices should be carefully cleaned and rinsed before calibration. See applicable AML SOP for additional guidance on glassware cleaning.

9.6 **Calibration Procedure:**

9.6.1 **Transfer pipettes:** Transfer pipettes are typically calibrated to deliver (TD) fixed volumes of liquids. The calibration of at least one pipette from each lot should be verified. Tare the weighing dish. Pipet the known volume of reagent water into the weighing dish using the pipette undergoing calibration check. Record the volume pipetted and the weight of water in the logbook (See Table III and IV) to 0.1-mg or four significant figures. Perform the measurement in triplicate. Record the temperature of the water in the logbook. Copy the density of water in air at that temperature from Table II. Calculate the mean, standard deviation and the percent relative standard deviation for the triplicate weight measurements. Calculate the mean observed volume of water from the known volume (W) of 1-g of water in air (Table II). Determine the difference (D) between the mean observed volume (OV) and the volume pipetted or true volume (TV) by subtracting TV from OV. Calculate the percent difference (%D) as described in section 8. If the %D for the mean is equal to or less than the tolerance specification, the pipette can be used without volume correction. The default tolerance specification for %D is ±1%. This procedure is also applicable to fixed volume autopipettors (e.g. Eppendorfs) and other devices that are similar in nature.

9.6.2 **Measuring pipettes and syringes:** Syringes and measuring pipettes have graduations that may allow variable volumes to be delivered. The calibration procedure for these devices is virtually identical to that of the transfer pipette with the exception that it should be repeated at three different volumes: 10%, 50% and 100% of the maximum volumes. For example, the volumes for a 1-ml syringe should be checked at 100-µl, 500-µl, and 1000-µl volume settings. This procedure is also applicable to variable volume autopipettors, dispensers and diluters.

9.6.3 **Graduated Cylinders:** Due to potential large volumes, weighing is not the recommended procedure to verify their calibration. To verify the calibration of 1000-ml graduated cylinders, fill a Class A 250-ml volumetric flask with reagent water to the mark. Pour the contents completely into the graduated cylinder. Record the volume. Repeat this procedure using two additional aliquots into the same cylinder. Use a similar procedure for volumetric cylinders of other sizes. Perform calculations as described in section 7.3 using 1.0000 for C, the correction factor. Class A measuring cylinders should be used to measure out samples used for analysis.

9.6.4 **Beakers, Erlenmeyers, etc:** These devices shall not be used either to contain or to deliver accurate volumes, which may enter into sample result calculations. Calibration of these devices is not required.

9.6.5 **Plastic sample digestion cups:** Fifty-ml plastic cups with graduation marks at 25-ml are used to measure aqueous samples for metals analysis. Calibration of the volumes shall be performed using one or more cups from each lot of sample cups before they are used to measure out samples. Tare the sample cup. Pour reagent water to the 25-ml mark. Record the weight to 0.1-mg. Pour additional reagent water to the 50-ml

mark. Record the weight again. Repeat this process in triplicate. Do not use these cups for measuring volumes other than those for which calibrations have been checked.

10 CALCULATIONS

10.1 Record water temperature, measured volumes and weights in a bound logbook that contain information as shown in Table III.

10.2 From triplicate weight measurements, calculate the mean observed weight (OW) and the standard deviation using standard laboratory procedures. Convert the mean observed weight into observed volume (OV) by multiplying it by the temperature correction factor (C) shown in Table II. Record the appropriate correction factor for the water temperature in the logbook.

10.3 Calculate the percent difference (%D) between OV and the true volume (TV) as follows:

$$OV = OW * C$$

$$\%D = \frac{|(OV-TV)| * 100}{TV}$$

11 QUALITY CONTROL

Calibrate one Class A device (if available) to validate the calibration process. The %D for the Class A device should lie within the stated tolerance (Table I and/or Appendix 2). A clean 10-ml Class A pipette is the preferred device to check the calibration procedure.

12 REFERENCES

12.1 Test Methods for Evaluating Solid Waste, Volume 1A: Laboratory Manual Physical/Chemical Methods, SW-846, EPA, Chapter 1, Quality Control, September 1994.

12.2 American Society for Testing (ASTM) Method Designation D5522-99a, Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid.

12.3 American Society for Testing (ASTM) Method Designation E542-00, Standard Practice for Calibration of Laboratory Volumetric Apparatus.

12.4 American Society for Testing (ASTM) Method Designation E694-99, Standard Specification for Laboratory Glass Apparatus.

12.5 American Society for Testing (ASTM) Method Designation E969-95, Standard Specification for Glass Volumetric (Transfer) pipettes.

12.6 American Society for Testing (ASTM) Method Designation E1154-89, Standard Specification for Piston or Plunger Operated Volumetric Apparatus.

12.7 American Society for Testing (ASTM) Method Designation E714-94, Standard Specification for Disposable Glass Serological pipettes.

12.8 American Society for Testing (ASTM) Method Designation E1293-94, Standard Specification for Glass Measuring pipettes, Reapproved 1998.

12.9 American Society for Testing (ASTM) Method Designation E1272-95, Standard Specification for Laboratory Glass Graduated Cylinders, Reapproved 1999.

12.10 American Society for Testing (ASTM) Method Designation E288-94, Standard Specification for Laboratory Glass Volumetric Flasks, Reapproved 1998.

12.11 American Society for Testing (ASTM) Method Designation E1878-97, Standard Specification for Laboratory Glass Volumetric Flasks, Special Use.

12.12 AML Standard Operating Procedures, Q-Water-RA.

12.13 EM 200-1-1, Validation of Analytical Chemistry Laboratories, US Army Corps of Engineers. July 1, 1994.

12.14 EM 200-1-3, Requirements for the Preparation of Sampling and Analysis Plans, Appendix I, US Army Corps of Engineers, February 1, 2001.

12.15 Department of Defense Quality Systems Manual (DoD QSM) for Environmental Laboratories, Final Version 2, June 2002.

Table I

Typical Tolerances for Volumetric Glassware

Capacity (ml)	Tolerance (ml) or % for AML limits			
	Volumetric flasks	Transfer pipettes	Burettes	AML Tolerance, %
2		0.006		≤ ± 1%
5		0.01	0.01	≤ ± 1%
10		0.02	0.02	≤ ± 1%
25	0.03	0.03	0.03	≤ ± 1%
50	0.05	0.05	0.05	≤ ± 1%
100	0.08	0.08	0.1	≤ ± 1%
200	0.10	0.10		≤ ± 1%
500	0.15			≤ ± 1%
1000	0.30			≤ ± 1%

See Appendix 2 for applicable ASTM Standards
See Text for detailed AML standards

Table II

Volume of 1-g of water in air at various Temperatures

Temperature (°C)	(ml/g)
10	1.0016
11	1.0017
12	1.0018
13	1.0019
14	1.0020
15	1.0021
16	1.0022
17	1.0023
18	1.0025
19	1.0026
20	1.0028
21	1.0030
22	1.0032
23	1.0034
24	1.0036
25	1.0038
26	1.0041
27	1.0043
28	1.0046
29	1.0048
30	1.0051

Table III - Volume Calibration Log - Class A Devices

Analytical Management Laboratories

Device to be calibrated:	
Device Label or ID, if any:	
Intended dedicated use, if any:	
Date:	
Temperature of water (T°C):	
Correction Factor (C) at T°C: (from Table II of the AML SOP)	

True Volume TV, ml	Observed Weight, OW (g) For triplicate measurements			Mean Weight	S. D.	RSD	Mean Volume	%Difference	AML Standard
	# 1	#2	# 3	OW (g)	(g)	%	OV, ml (C*OW)	|(OV-TV)| / TV in percent	Tolerance %
									≤ ± 1%
									≤ ± 1%
									≤ ± 1%
									≤ ± 1%
									≤ ± 1%
									≤ ± 1%
									≤ ± 1%
									≤ ± 1%
									≤ ± 1%
									≤ ± 1%
									≤ ± 1%
									≤ ± 1%
									≤ ± 1%
									≤ ± 1%
									≤ ± 1%

Table IV - Volume Calibration Log - non-Class A Devices

Analytical Management Laboratories

Device to be calibrated:	
Device Label or ID, if any:	
Intended dedicated use, if any:	
Date:	
Temperature of water (T°C):	
Correction Factor (C) at T°C: (from Table II of the AML SOP)	

True Volume TV, ml	Observed Weight, OW (g) For triplicate measurements			Mean Weight	S. D.	RSD	Mean Volume	%Difference	AML Standard
	# 1	#2	# 3	OW (g)	(g)	%	OV, ml (C*OW)	|(OV-TV)| / TV in percent	Tolerance %
Class A 10-mL									**≤ ± 1% (control)**
									≤ ± 3%
									≤ ± 3%
									≤ ± 3%
									≤ ± 3%
									≤ ± 3%
									≤ ± 3%
									≤ ± 3%
									≤ ± 3%
									≤ ± 3%
									≤ ± 3%
									≤ ± 3%
									≤ ± 3%
									≤ ± 3%
									≤ ± 3%

Table V
List of Available Appendices
(Not included in this document)

Appendix Number	Appendix Topic
1	Example of Inscriptions for Volumetric Apparatus
2	ASTM Specifications for the Tolerance of Volumetric Apparatus E969 - Glass Transfer Pipettes
3	ASTM Specifications for the Tolerance of Volumetric Apparatus E1293 - Glass Measuring Pipettes
4	ASTM Specifications for the Tolerance of Volumetric Apparatus E714 - Glass Disposable serological Pipettes
5	ASTM Specifications for the Tolerance of Volumetric Apparatus E288 - Volumetric Flasks
6	ASTM Specifications for the Tolerance of Volumetric Apparatus E1272 - Graduated Cylinders

NOTE:
Appendices cited in this document contain graphics, hand-written notes, computer printouts prepared using specialized software, and other information that are intended to provide additional guidance to AML laboratory personnel. The appendices to this SOP are dynamic laboratory tools that are updated more frequently than the referenced SOP documents. The appendices are restricted to circulation among laboratory personnel only. Due to the size and nature of the appendices, the SOP document is provided to A/E firms and government agencies (potentially for use in the preparation of project documents) in hardcopy and/or electronic format without the appendices. The appendices may be available in hardcopy form or as portable document electronic files (pdf).

STANDARD OPERATING PROCEDURES

No. Q-WasteMgmt-RE

WASTE MANAGEMENT AND DISPOSAL INCLUDING SAMPLE DISPOSAL

October 31, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) is based on Hazardous Waste Management Guide for Laboratories, a publication sponsored by the American Council of Independent Laboratories (ACIL). This SOP is used for the following purposes: to minimize laboratory generated waste; to segregate and manage potentially hazardous waste from non-hazardous waste; and to dispose waste and wastewater in accordance with applicable federal, state and local rules and regulations. This SOP shall be used in conjunction with management directives, if any, to address changes that may be necessary for rapid compliance with regulatory changes.

2.0 SUMMARY OF THE PROCEDURE

Wastes generated by the laboratory are categorized based on the knowledge of underlying materials and/or constituents. Wastes that cannot be disposed by pouring into the drain or pitched into a sanitary waste receptacle are containerized and handled appropriately.

3.0 WASTE CATEGORIES

Wastes are categorized as described in Table I. Please see appropriate short waste management form (Appendix 1) for the type of wastestream, source, characteristics, management and disposal options and other details.

4.0 WASTE COLLECTION PROCEDURES

Waste generated by the laboratory and fitting one or more of the categories described above will be collected, handled, managed and disposed using one of the waste disposal methods described in TABLE II. Details of the disposal procedure are described in Appendix 1.

5.0 WASTE STORAGE CONTAINERS

Drums and receptacles potentially containing hazardous waste are located in a secured storage shed immediately behind the laboratory building. Containers for accumulating small volumes of wastestreams to be disposed of as shown in Table II are located within the laboratory building. The waste disposal coordinator or other trained personnel are responsible for handling and transferring waste from the laboratory building to the storage shed.

6.0 SAMPLE WASTE CHARCTERIZTION AND DISPOSAL PROCEDURES

For initial (within the laboratory) disposal of unused samples, the hazardous characteristics of the sample waste is established from available test results and observations made by laboratory personnel. It is based on the assumption that tests ordered by the client are sufficient to characterize samples for parameters of concern. Since this assumption is not necessarily true, visual observations (odor, presence of oil and/or organic solvents, etc.) will be used to supplement test data. However, the laboratory shall not run any additional tests on client samples that have not been authorized by the client. The ultimate disposal of accumulated soil will be determined from the hazardous characteristics of the composite sample waste(s) from multiple client sources.

The sample is screened as potentially hazardous if it meets one or more of the following criteria:

(a) It is a sample of a listed (F, K, U, etc.) hazardous waste (if information is available).
(b) It is reactive (water-reactive, detonates, D003) or contains high levels of releasable cyanide (250 mg HCN/kg) or sulfide (500 mg H_2S/kg).
(c) It is ignitable (D001, flashpoint less than 140°F or 60°C).
(d) It is corrosive (D002, pH <2.0 or >12.5) or corrodes steel at a high rate (>6.35 mm/year).
(e) It is toxic (D004 to D043) because it has one or more of the 40 TC constituents (Table III) in excess of the regulatory limits shown either in the TCLP leachates (if available) or twenty times these levels in the whole sample.
(f) It has high levels of PCBs (>50 mg/kg).
(g) It contains dioxins and/or furans (>1.0 ug/kg TEF).
(h) It appears to have the potential to meet one of the above listed criteria.

Even if unused sample(s) from one source exhibits hazardous characteristics, there is potential for accumulated unused samples (waste) to be non-hazardous. The presence or absence of LDR constituents (40 CFR 268.41) other than the 40 TC constituents shall not be used to screen samples for potential hazardous characteristics.

For each AML project, the Operations Manager (or a delegate) will provide instructions for the sample disposal on a modified COC form, providing direction and time frame for the initial disposal of each sample. Unused samples will be disposed accordingly. The actual disposal of unused samples will be performed either by the Sample Custodian or a qualified technician using information provided by the Operations Manager. Person disposing samples will sign and date either this form or the Sample Tracking Form used for internal custody of samples with comments describing exceptions, etc.

7.0 NON-SAMPLE WASTE DISPOSAL PROCEDURES

Laboratory wastes other than unused samples are laboratory process wastes with potential for hazardous characteristics. Appendix 1 contains short forms and detailed directions for handling various wastestreams. Table II contains direction for the accumulation and disposal of these wastes in accordance with applicable rules and regulations. The waste disposal coordinator or other trained personnel are responsible for handling and transferring waste from the laboratory building to the storage shed.

8.0 RECORDKEEPING AND REPORTING PROCEDURES

The waste disposal coordinator is responsible for maintaining the originals and/or copies of the Hazardous Waste Manifests, Land Disposal Restriction (LDR) Forms, Waste Profile Sheets and other applicable reports.

9.0 QUALITY CONTROL AND AUDITING PROCEDURES

The Quality Assurance Officer is responsible for conducting periodic audits of the Waste disposal procedure to assure compliance with applicable laws and regulations.

TABLE I

No.	WASTE DESCRIPTION	HAZARD RATING
W01	Household waste	Non-hazardous waste
W02	Empty containers	Non-hazardous waste
W03	Residues of hazardous waste in empty containers	Non-hazardous waste
W04	Unused non-hazardous samples-containerized or otherwise	Non-hazardous waste
W05	Extracted soil samples	Non-hazardous waste
W06	Extracted common drying agents	Non-hazardous waste
W07	Broken glass	Non-hazardous waste
W08	Hazardous soil samples	Hazardous waste
W09	Extracted water samples (CH_2Cl_2)	Non-hazardous wastewater
W10	Extracted water samples (Hexane)	Potentially hazardous wastewater
W11	Rinsewater from cleaning glassware, bottles, etc.	Non-hazardous wastewater
W12	Unused fuel samples	Recyclable waste
W13	Used Pump Oil (mineral oil based)	Recyclable waste
W14	Organic sample extracts	Hazardous
W15	Spent organic solvents	Hazardous
W16	Sample extracts in 2-mL vials	Hazardous
W17	Unused compressed gases	Potentially hazardous
W18	Foreign soil	Potentially biohazardous
W19	Biohazardous materials	Potentially biohazardous
W20	PCBs containing material	Hazardous characteristic waste
W21	Dioxin containing material	Acutely hazardous characteristic waste
W22	Unused chemicals	Potentially hazardous labpackable waste
W23	Digestates from metals analysis	Hazardous characteristic waste
W24	TKN & COD test residuals	Hazardous waste
W25	To be Identified	

TABLE II

#	DISPOSAL METHOD	WASTE CATEGORIES	COMMENTS
1	Trash Dumpster	W01, W02, W03, W04, W05, W06, W07, and applicable W18 and W19	Contact Waste Coordinator if in doubt.
2	Soil HazWaste Drum	W08 and applicable W18 and W19	Black open-top drums. Keep the lid on at all times.
3	PCB Waste	W20	Black 5-gallon containers.
4	Non-PCB Oil/Petroleum	W12 and W13	Yellow 5-gallon containers.
5	Chlorinated Solvent Waste	W14 and W15	Screw-cap 55 Gallon drums. Keep drums closed at all times.
6	Acid/Metals Waste	W23	55 gallon plastic drums
7	Mercury Containing Waste	W24	5 gallon containers
8	GC Vials (Solid/Liquid)	W16	Black open-top drums. Keep the lid on at all times.
9	Misc. Lab Pack	W21 and W22	Contact Waste Coordinator for extended storage of unused samples and chemicals.
10	Basic Waste		
11	Laboratory Sink/City Sewer	W09, W10 and W11	Neutralization and/or purging may be necessary for some wastestreams.
12	Return to Client/Vendor	W17	Coordinate with vendor.

TABLE III

Maximum Concentration of Contaminants for the Toxicity Characteristics

EPA HW Number	Contaminant	CAS Number	Regulatory Level (mg/L) Leachate	Guidance Level (mg/kg) Total
METALS:				
D004	Arsenic.....	7440-38-2	5.0	100
D005	Barium.....	7440-39-3	100.0	2000
D006	Cadmium.....	7440-43-9	1.0	20
D007	Chromium.....	7440-47-3	5.0.	100
D008	Lead.....	7439-92-1	5.0	100
D009	Mercury.....	7439-97-6	0.2	4.0
D010	Selenium.....	7782-49-2	1.0	20
D011	Silver.....	7440-22-4	5.0	100
VOCs:				
D018	Benzene.....	71-43-2	0.5	10
D019	Carbon tetrachloride.....	56-23-5	0.5	10
D021	Chlorobenzene.....	108-90-7	100.0	2000
D022	Chloroform.....	67-66-3	6.0	120
D028	1,2-Dichloroethane.....	107-06-2	0.5	10
D029	1,1-Dichloroethylene.....	75-35-4	0.7	14
D035	Methyl ethyl ketone.....	78-93-3	200.0	4000
D039	Tetrachloroethylene.....	127-18-4	0.7	14
D040	Trichloroethylene.....	79-01-6	0.5	10
D043	Vinyl chloride.....	75-01-4	0.2	4.0
SVOCs:				
D023	o-Cresol.....	95-48-7	200.0	4000
D024	m-Cresol.....	108-39-4	200.0	4000
D025	p-Cresol.....	106-44-5	200.0	4000
D026	Cresol, Total.....	NA	200.0	4000
D027	1,4-Dichlorobenzene.....	106-46-7	7.5	150
D030	2,4-Dinitrotoluene.....	121-14-2	>0.13	2.6
D032	Hexachlorobenzene.....	118-74-1	>0.13	2.6
D033	Hexachlorobutadiene.....	87-68-3	0.5	10
D034	Hexachloroethane.....	67-72-1	3.0	60
D036	Nitrobenzene.....	98-95-3	2.0	40
D037	Pentachlorophenol.....	87-86-5	100.0	2000
D038	Pyridine.....	110-86-1	>5.0	100
D041	2,4,5-Trichlorophenol.....	95-95-4	400.0	8000
D042	2,4,6-Trichlorophenol.....	88-06-2	2.0	40

TABLE III (contd.)

Maximum Concentration of Contaminants for the Toxicity Characteristics

EPA HW Number	Contaminant	CAS Number	Regulatory Level (mg/L) Leachate	Guidance Level (mg/kg) Total
Pesticides:				
D020	Chlordane.....	57-74-9	0.03	0.6
D012	Endrin.....	72-20-8	0.02	0.4
D031	Heptachlor (and its epoxide)	76-44-8	0.008	0.16
D013	Lindane.....	58-89-9	0.4	8.0
D014	Methoxychlor.....	72-43-5	10.0	200
D015	Toxaphene.....	8001-35-2	0.5	10
Herbicides:				
D016	2,4-D.....	94-75-7	10.0	200
D017	2,4,5-TP (Silvex).....	93-72-1	1.0	20

APPENDIX – 1

WASTE MANAGEMENT FORMS

Waste Management Form

ITEM	DESCRIPTION
Item Number	W01
Wastestream and Source	Household Waste: trash from office areas, garbage, paper and miscellaneous solid waste; waste of a similar nature from laboratory areas including gloves, paper towels, weighing boats, aluminum foil, etc.
Hazardous Characteristics	Non-hazardous; similar in characteristics to waste profile # WS97000072.
On-Site Management Options:	
Sewarable? ……...	……Not Applicable
Sanitary Landfillable? …….	……Yes; place in trash receptacles which are emptied into dumpsters; picked up by local sanitation department.
Other: ……..	……Not Applicable
Off-Site Management Options:	Sanitary Landfill
Transportation Requirements:	
DOT shipping name: …….	…….. Non-RCRA Material, Solid (Soil/Glass/Misc.)
Hazard Class: ……	……..
UN/NA No.: …….……	……..
EPA Waste No.: ……...	……..
Physical State: ……..	……..
Container Type: ……	……..
Other: ……	……..

LDR information:

Treatment Alternatives:
Treatment Alternative 1: ……..

Treatment Alternative 2: ……..

Treatment Alternative 3: ……..

Recycling Alternatives:
Recycling Alternative 1: ……..

Recycling Alternative 2: ……..

Recycling Alternative 3: ……..

Disposal Alternatives:
Disposal Alternative 1: ……

Disposal Alternative 2: ……

Disposal Alternative 3: ……

General Comments:

Waste Management Form

ITEM	DESCRIPTION
Item Number(s)	W02, W03 and W04
Wastestream and Source	W02: Empty containers: Includes unused or used containers (triple-rinsed for strong acids/bases) not containing any sample, reagents, etc. W03: Empty containers containing residues of hazardous materials/waste. W04: Non-hazardous samples, containerized or otherwise. See general comments for details.
Hazardous Characteristics	Non-hazardous if conditions described in general comments are met; similar in characteristics to waste profile # WS97000072.
On-Site Management Options:	
Sewarable? ……..	……NA
Sanitary Landfillable? …….	……Yes; place in trash receptacles and emptied into dumpsters; package broken glass in cardboard boxes; picked up by local sanitation department.
Other: ……..	……Not Applicable
Off-Site Management Options:	Sanitary Landfill. See general comments.
Transportation Requirements:	
DOT shipping name: …….	…….. Non-RCRA Material, solid (soil/glass)
Hazard Class: ……	……..
UN/NA No.: …….……	……..
EPA Waste No.: ……...	……..
Physical State: ……..	……..
Container Type: ……	……..
Other: ……	……..
LDR information:	
Treatment Alternatives:	
Treatment Alternative 1: ……..	
Treatment Alternative 2: ……..	
Treatment Alternative 3: ……..	
Recycling Alternatives:	
Recycling Alternative 1: ……..	
Recycling Alternative 2: ……..	
Recycling Alternative 3: ……..	
Disposal Alternatives:	
Disposal Alternative 1: ……	
Disposal Alternative 2: ……	
Disposal Alternative 3: ……	
General Comments:	Adhere to 40CFR Part 261.7(a)(1): Remove all hazardous material/waste that can be removed using practices commonly employed to remove materials from that type of container so that: no more than 2.5 centimeters (one inch) of residue remains on the bottom of the container or inner liner; or no more than 3 percent by weight of the total capacity of the container remains if the container is less than or equal to 110 gallons in size; or no more than 0.3 percent by weight remains for containers greater than 110 gallons in size. Cross-out client information using dark indelible ink or remove sample label prior to disposing containers with unused hazardous soil.

Waste Management Form

ITEM	DESCRIPTION
Item Number(s)	W05 and W06
Wastestream and Source	W05: Soil samples after solvent extraction W06: Drying agents (anhydrous sodium sulfate, calcium chloride, etc.)
Hazardous Characteristics	Non-hazardous; similar in characteristics to waste profile # WS97000072. See general comments for details
On-Site Management Options:	
Sewarable? ……..	……NA
Sanitary Landfillable? …….	…… Yes; place in the hood overnight for the solvent to evaporate off and empty into trash receptacles.
Other: ……..	……NA
Off-Site Management Options:	Sanitary Landfill after solvent removal. See general comments.
Transportation Requirements:	
DOT shipping name: …….	…….. Non-RCRA Material, solid (soil/glass/Misc.)
Hazard Class: ……	……..
UN/NA No.: ……	……..
EPA Waste No.: ……...	……..
Physical State: ……..	……..
Container Type: ……	……..
Other: ……	……..
LDR information:	
Treatment Alternatives:	
Treatment Alternative 1: ……..	
Treatment Alternative 2: ……..	
Treatment Alternative 3: ……..	
Recycling Alternatives:	
Recycling Alternative 1: ……..	
Recycling Alternative 2: ……..	
Recycling Alternative 3: ……..	
Disposal Alternatives:	
Disposal Alternative 1: ……	
Disposal Alternative 2: ……	
Disposal Alternative 3: ……	
General Comments:	Extracted soil and drying agents may contain small amounts of adhered solvents. Remove these solvents by placing them for approx. 8 hrs. in an open container in the hood with the fans running. Treat the residues as non-hazardous solid waste.

Waste Management Form

ITEM	DESCRIPTION
Item Number	W07
Wastestream and Source	Broken glass from the laboratory. Includes used/unused Pasteur pipettes, broken glass in general and other sharp objects.
Hazardous Characteristics	Non-hazardous; similar in characteristics to waste profile # WS97000072. See general comments for W02, W03 and W04 for guidance on adhering hazardous material/waste.
On-Site Management Options:	
Sewarable? ……...	……NA
Sanitary Landfillable? …….	……Yes. See general comments.
Other: ……..	……NA
Off-Site Management Options:	Sanitary Landfill. See general comments.
Transportation Requirements:	
DOT shipping name: …….	…….. Non-RCRA Material, solid (glass)
Hazard Class: ……	……..
UN/NA No.: …….……	……..
EPA Waste No.: ……...	……..
Physical State: ……..	……..
Container Type: ……	……..
Other: ……	……..
LDR information:	
Treatment Alternatives:	
Treatment Alternative 1: ……..	
Treatment Alternative 2: ……..	
Treatment Alternative 3: ……..	
Recycling Alternatives:	
Recycling Alternative 1: ……..	
Recycling Alternative 2: ……..	
Recycling Alternative 3: ……..	
Disposal Alternatives:	
Disposal Alternative 1: ……	
Disposal Alternative 2: ……	
Disposal Alternative 3: ……	
General Comments:	Collect waste in metal containers. Empty with care into cardboard boxes. Tape them shut and dispose boxes into dumpsters.

Waste Management Form

ITEM	DESCRIPTION
Item Number	W08
Wastestream and Source	Unused hazardous soil samples in containers.
Hazardous Characteristics	Hazardous
On-Site Management Options:	
Sewarable? ……...	……NA
Sanitary Landfillable? …….	……No.
Other: ……..	……Containerized in 55-gallon open top drums.
Off-Site Management Options:	Collect and manage for periodic off-site disposal.
Transportation Requirements:	
DOT shipping name: …….	……..
Hazard Class: ……	……..
UN/NA No.: …….……	……..
EPA Waste No.: ……...	……..
Physical State: ……..	……..
Container Type: ……	……..
Other: ……	……..
LDR information:	
Treatment Alternatives:	
Treatment Alternative 1: ……..	
Treatment Alternative 2: ……..	
Treatment Alternative 3: ……..	
Recycling Alternatives:	
Recycling Alternative 1: ……..	
Recycling Alternative 2: ……..	
Recycling Alternative 3: ……..	
Disposal Alternatives:	
Disposal Alternative 1: ……	
Disposal Alternative 2: ……	
Disposal Alternative 3: ……	
General Comments:	Empty smaller amounts of samples from large containers either directly into the 55-gallon drum or into other bottles. Dispose smaller and full/nearly full containers directly into the drum. Characterize a representative sample to determine disposal options.

Waste Management Form

ITEM	DESCRIPTION
Item Number(s)	W09 and W10
Wastestream and Source	W09: Water samples after extraction with methylene chloride and unused water samples. W10: Water samples after extraction with hexane.
Hazardous Characteristics	May contain low levels of methylene chloride or hexane (flammable solvent). Non-hazardous waste requiring either no laboratory processing (W09) or some processing (W10) prior to sewer disposal.
On-Site Management Options:	
Sewarable? ……..	……Yes-methylene chloride contaminated water without treatment. ……Yes-hexane contaminated water after purging to remove hexane.
Sanitary Landfillable? …….	……NA
Other: ……..	……NA
Off-Site Management Options:	Collect, purge and dispose into city sewer.
Transportation Requirements:	
DOT shipping name: …….	……..NA
Hazard Class: ……	……..
UN/NA No.: ……	……..
EPA Waste No.: ……...	……..
Physical State: ……..	……..
Container Type: ……	……..
Other: ……	……..
LDR information:	
Treatment Alternatives:	
Treatment Alternative 1: ……..	
Treatment Alternative 2: ……..	
Treatment Alternative 3: ……..	
Recycling Alternatives:	
Recycling Alternative 1: ……..	
Recycling Alternative 2: ……..	
Recycling Alternative 3: ……..	
Disposal Alternatives:	
Disposal Alternative 1: ……	
Disposal Alternative 2: ……	
Disposal Alternative 3: ……	
General Comments:	Collect all extracted water containing hexane (optional for methylene chloride containing water) into 5-gallon containers in a hood. Purge them overnight with air with the fans running and empty the residual water into sink. Most unused water samples can be discharged into the laboratory sink (city sewer).

Waste Management Form

ITEM	DESCRIPTION
Item Number	W11
Wastestream and Source	Rinsewater from cleaning glassware, bottles, etc.
Hazardous Characteristics	Non-hazardous. See general comments at the bottom for exceptions.
On-Site Management Options:	
Sewarable? ……..	……Yes.
Sanitary Landfillable? …….	……NA
Other: ……..	……NA
Off-Site Management Options:	Empty into sink (city sewer). See general comments at the bottom.
Transportation Requirements:	
DOT shipping name: …….	……..NA
Hazard Class: ……	……..
UN/NA No.: …….……	……..
EPA Waste No.: ……...	……..
Physical State: ……..	……..
Container Type: ……	……..
Other: ……	……..
LDR information:	
Treatment Alternatives:	
Treatment Alternative 1: ……..	
Treatment Alternative 2: ……..	
Treatment Alternative 3: ……..	
Recycling Alternatives:	
Recycling Alternative 1: ……..	
Recycling Alternative 2: ……..	
Recycling Alternative 3: ……..	
Disposal Alternatives:	
Disposal Alternative 1: ……	
Disposal Alternative 2: ……	
Disposal Alternative 3: ……	
General Comments:	Exception: Empty strong acid or strong base bottles completely by consolidation of the contents into new containers. Collect first washings of these containers and dispose them as acid/base waste as appropriate. See metal digestates (W25) for details.

Waste Management Form

ITEM	DESCRIPTION
Item Number(s)	W12 and W13
Wastestream and Source	W12: unused fuel-containing samples from UST sites (e.g. tank contents). W13: used mineral oil based pump oil (GC/MS).
Hazardous Characteristics	Non-hazardous; similar in characteristics to waste profile # WS97000858 and WS-98000016. Pump oil may contain small amounts of VOCs and SVOCs at levels that are not of concern. Recyclable petroleum material.
On-Site Management Options:	
Sewarable? ……...	……No.
Sanitary Landfillable? …….	……No.
Other: ……..	……Yes; Collect into special containers for off-site disposal.
Off-Site Management Options:	Off-site disposal. Recycling as fuel.
Transportation Requirements:	
DOT shipping name: …….	…….. Non-RCRA Material, Liquid (used oil)
Hazard Class: ……	……..NA
UN/NA No.: …….……	……..NA
EPA Waste No.: ……...	……..None
Physical State: ……..	……..Liquid
Container Type: ……	……..Drum
Other: ……	……..
LDR information:	
Treatment Alternatives:	
Treatment Alternative 1: ……..	
Treatment Alternative 2: ……..	
Treatment Alternative 3: ……..	
Recycling Alternatives:	
Recycling Alternative 1: ……..	…. Used as fuel in cement kilns, boilers and industrial furnaces.
Recycling Alternative 2: ……..	
Recycling Alternative 3: ……..	
Disposal Alternatives:	
Disposal Alternative 1: ……	…. Contact Waste Hauler.
Disposal Alternative 2: ……	
Disposal Alternative 3: ……	
General Comments:	Do **NOT** mix used hydrocarbon based fuels and pump oils with potentially hazardous liquid materials such as PCB oils, silicone oils, etc.

Waste Management Form

ITEM	DESCRIPTION
Item Number(s)	W14 and W15
Wastestream and Source	W14: Organic sample extracts in screw-cap containers larger than 2-mL. W15: Used/recovered but unusable organic solvents.
Hazardous Characteristics	Hazardous. Listed (F) RCRA hazardous waste with hazardous waste characteristics (ignitability and toxicity).
On-Site Management Options:	
Sewarable? ……...	……No.
Sanitary Landfillable? …….	……No.
Other: ……..	…… Collect, containerize and manage for off-site disposal. See general comments.
Off-Site Management Options:	Off-site disposal.
Transportation Requirements:	
DOT shipping name: …….	……..RQ Waste Flammable Liquid (Acetone/DCM)
Hazard Class: ……	……..3
UN/NA No.: ……..……	……..UN 1593, UN1993
EPA Waste No.: ……...	……..F002, F003, D001
Physical State: ……..	……..Liquid
Container Type: ……	……..Group 111-closed top drum
Other: ……	……..Label as “Keep away from food”.
LDR information:	Prohibited.
Treatment Alternatives:	
Treatment Alternative 1: ……..	Incineration
Treatment Alternative 2: ……..	
Treatment Alternative 3: ……..	
Recycling Alternatives:	
Recycling Alternative 1: ……..	As fuel in cement kilns, boilers and industrial furnaces if BTU levels are high enough and % chlorine are low enough.
Recycling Alternative 2: ……..	
Recycling Alternative 3: ……..	
Disposal Alternatives:	
Disposal Alternative 1: ……	Contact Waste Hauler.
Disposal Alternative 2: ……	
Disposal Alternative 3: ……	
General Comments:	Do **NOT** mix W14 and W15 (chlorinated solvent waste) with hydrocarbon wastes, PCB-containing solvent wastes and other types of similar liquid materials such as PCB oils, silicone oils, etc. Empty solvents into designated containers and discard empty vials, bottles, etc. as W01 solid waste. Remove solvents to the extent possible by leaving empty containers in an operating hood.

Waste Management Form

ITEM	DESCRIPTION
Item Number	W16
Wastestream and Source	Sample extracts in 2-mL GC vials or smaller containers. Unused or expired organic standard solutions in similar containers with the exception of dioxin-containing standards and sample extracts.
Hazardous Characteristics	Hazardous. Listed (F) RCRA hazardous waste with hazardous waste characteristics (ignitability and toxicity).
On-Site Management Options:	
Sewarable? ……...	……No
Sanitary Landfillable? …….	……No
Other: ……..	…… Collect, containerize and manage for off-site disposal. See general comments.
Off-Site Management Options:	Off-site disposal.
Transportation Requirements:	
DOT shipping name: …….	……..RQ Waste Flammable Liquid, solid (Acetone/DCM, glass)
Hazard Class: ……	……..3
UN/NA No.: …….……	……..UN 1593, UN1993
EPA Waste No.: ……...	……..F002, F003, D001
Physical State: ……..	……..Liquid
Container Type: ……	……..Group 111-closed top drum
Other: ……	……..Label as “Keep away from food”.
LDR information:	Prohibited.
Treatment Alternatives:	
Treatment Alternative 1: ……..	Incineration.
Treatment Alternative 2: ……..	
Treatment Alternative 3: ……..	
Recycling Alternatives:	
Recycling Alternative 1: ……..	
Recycling Alternative 2: ……..	
Recycling Alternative 3: ……..	
Disposal Alternatives:	
Disposal Alternative 1: ……	Contact Waste Hauler.
Disposal Alternative 2: ……	
Disposal Alternative 3: ……	
General Comments:	Do **NOT** try to open either the crimp-top or the screw-cap vials to empty solutions. Dispose sealed intact vials (not leaking) into marked receptacles. Do **NOT** include PCB-containing or dioxin-containing materials in to the W16 wastestream.

Waste Management Form

ITEM	DESCRIPTION
Item Number	W17
Wastestream and Source	Unused compressed gases
Hazardous Characteristics	W17 is not a solid waste. Potential physical hazard; potential for flammable hazard.
On-Site Management Options:	
Sewarable? ……...	……No
Sanitary Landfillable? …….	……No
Other: ……..	……Yes. Return used, unusable or unused (but no longer required) compressed gas cylinder to the vendor.
Off-Site Management Options:	NA
Transportation Requirements:	
DOT shipping name: …….	……..Non-RCRA Material
Hazard Class: ……	……..
UN/NA No.: …….……	……..
EPA Waste No.: ……...	……..
Physical State: ……..	……..
Container Type: ……	……..
Other: ……	……..
LDR information:	
Treatment Alternatives:	
Treatment Alternative 1: ……..	
Treatment Alternative 2: ……..	
Treatment Alternative 3: ……..	
Recycling Alternatives:	
Recycling Alternative 1: ……..	
Recycling Alternative 2: ……..	
Recycling Alternative 3: ……..	
Disposal Alternatives:	
Disposal Alternative 1: ……	
Disposal Alternative 2: ……	
Disposal Alternative 3: ……	
General Comments:	NOTE: Manage empty, partial and full cylinders in accordance with applicable laboratory chemical hygiene plans.

Waste Management Form

ITEM	DESCRIPTION
Item Number(s)	W18 and W19
Wastestream and Source	W18: Foreign and quarantined domestic soil. W19: Biohazardous materials (tissues, body fluids, etc.)
Hazardous Characteristics	Potential for biohazard. Potential for hazardous characteristics. After biohazard is removed, use other applicable waste management principles.
On-Site Management Options:	
Sewarable? ……..	……No.
Sanitary Landfillable? …….	……Yes-treated non-hazardous soil. No-treated hazardous soil.
Other: ……..	……Autoclave all unused soil in accordance with applicable SOP. Manage and dispose autoclaved soil as described for unused soil samples.
Off-Site Management Options:	
Transportation Requirements:	
DOT shipping name: …….	……..
Hazard Class: ……	……..
UN/NA No.: ……	……..
EPA Waste No.: ……...	……..
Physical State: ……..	……..
Container Type: ……	……..
Other: ……	……..
LDR information:	
Treatment Alternatives:	
Treatment Alternative 1: ……..	Pretreatment: Autoclave to destroy endemic pests, pathogens, parasites and other non-endemic factors that are not natural to the area.
Treatment Alternative 2: ……..	
Treatment Alternative 3: ……..	
Recycling Alternatives:	
Recycling Alternative 1: ……..	
Recycling Alternative 2: ……..	
Recycling Alternative 3: ……..	
Disposal Alternatives:	
Disposal Alternative 1: ……	
Disposal Alternative 2: ……	
Disposal Alternative 3: ……	
General Comments:	See applicable SOPs for autoclaving foreign soil. See waste management forms for wastestreams W04 and W08 for disposing autoclaved soil.

Waste Management Form

ITEM	DESCRIPTION
Item Number	W20
Wastestream and Source	PCB-containing materials. Unknown oily (potentially hazardous) materials, mixed with sulfuric acid (corrosive) and hexane (flammable)
Hazardous Characteristics	Hazardous with characteristics similar to waste profile # WS-980000020.
On-Site Management Options:	
Sewarable? ……..	……No
Sanitary Landfillable? …….	……No
Other: ……..	……Accumulate and dispose as hazardous waste.
Off-Site Management Options:	Off-site disposal
Transportation Requirements:	
DOT shipping name: …….	……..RQ Waste Corrosive Flammable Liquid, Acidic, Inorganic, n.o.s (glass/oil/acid)
Hazard Class: ……	……..8
UN/NA No.: …….……	……..UN3264
EPA Waste No.: ……...	……..D001, D002, D003
Physical State: ……..	……..
Container Type: ……	……..Drum, PG II
Other: ……	……..
LDR information:	
Treatment Alternatives:	
Treatment Alternative 1: ……..	
Treatment Alternative 2: ……..	
Treatment Alternative 3: ……..	
Recycling Alternatives:	
Recycling Alternative 1: ……..	
Recycling Alternative 2: ……..	
Recycling Alternative 3: ……..	
Disposal Alternatives:	
Disposal Alternative 1: ……	
Disposal Alternative 2: ……	
Disposal Alternative 3: ……	
General Comments:	Segregate PCB containing materials from non-PCB wastes, etc. Dispose PCB standards, GC vials, sulfuric acid used in cleanup, excess PCB-containing soil samples, etc. as PCB wastes.

Waste Management Form

ITEM	DESCRIPTION
Item Number	W21
Wastestream and Source	Dioxin containing samples and dioxin/furan standards
Hazardous Characteristics	Acute hazardous waste
On-Site Management Options:	
Sewarable? ……..	……No
Sanitary Landfillable? …….	……No
Other: ……..	……Accumulate as special hazardous waste for off-site disposal.
Off-Site Management Options:	Off-site disposal.
Transportation Requirements:	
DOT shipping name: …….	……..
Hazard Class: ……	……..
UN/NA No.: …….……	……..
EPA Waste No.: ……...	……..
Physical State: ……..	……..
Container Type: ……	……..
Other: ……	……..
LDR information:	
Treatment Alternatives:	
Treatment Alternative 1: ……..	Incineration
Treatment Alternative 2: ……..	
Treatment Alternative 3: ……..	
Recycling Alternatives:	
Recycling Alternative 1: ……..	
Recycling Alternative 2: ……..	
Recycling Alternative 3: ……..	
Disposal Alternatives:	
Disposal Alternative 1: ……	
Disposal Alternative 2: ……	
Disposal Alternative 3: ……	
General Comments:	Maintain unused samples as samples (not waste). Maintain unused standards as chemicals (not waste) in a secure area. Dispose when sufficient quantities are available for lab packing.

Waste Management Form

ITEM	DESCRIPTION
Item Number	W22
Wastestream and Source	Unused chemicals.
Hazardous Characteristics	Potentially hazardous. Check P list.
On-Site Management Options:	
Sewarable? ……..	……No
Sanitary Landfillable? …….	……May be for non-P list chemicals if they are determined to be non-toxic.
Other: ……..	……See comments. Lab pack for off-site disposal.
Off-Site Management Options:	Off-site disposal.
Transportation Requirements:	
DOT shipping name: …….	……..
Hazard Class: ……	……..
UN/NA No.: …….……	……..
EPA Waste No.: ……...	……..
Physical State: ……..	……..
Container Type: ……	……..
Other: ……	……..
LDR information:	
Treatment Alternatives:	
Treatment Alternative 1: ……..	
Treatment Alternative 2: ……..	
Treatment Alternative 3: ……..	
Recycling Alternatives:	
Recycling Alternative 1: ……..	
Recycling Alternative 2: ……..	
Recycling Alternative 3: ……..	
Disposal Alternatives:	
Disposal Alternative 1: ……	
Disposal Alternative 2: ……	
Disposal Alternative 3: ……	
General Comments:	Maintain stock of unused chemicals as economically useful reagents until they are no longer needed. Lab pack expired chemicals periodically for off-site disposal.

Waste Management Form

ITEM	DESCRIPTION
Item Number	W23
Wastestream and Source	Metal digestates
Hazardous Characteristics	Corrosivity and metals toxicity; similar in characteristics to waste profiles WS970000858 and WS98000018
On-Site Management Options:	
Sewarable? ……..	……No
Sanitary Landfillable? …….	……No
Other: ……..	……Accumulate in 55-gallon plastic drums for off-site disposal.
Off-Site Management Options:	Off-site disposal
Transportation Requirements:	
DOT shipping name: …….	……..RQ Waste Corrosive Liquid, Acidic, Inorganic, n.o.s., (nitric acid)
Hazard Class: ……	……..8
UN/NA No.: …….……	……..UN3264
EPA Waste No.: ……...	……..D002, D004, D005, D006, D007, D008, D010, D011
Physical State: ……..	……..liquid
Container Type: ……	……..Drum (PG I)
Other: ……	……..
LDR information:	
Treatment Alternatives:	
Treatment Alternative 1: ……..	
Treatment Alternative 2: ……..	
Treatment Alternative 3: ……..	
Recycling Alternatives:	
Recycling Alternative 1: ……..	
Recycling Alternative 2: ……..	
Recycling Alternative 3: ……..	
Disposal Alternatives:	
Disposal Alternative 1: ……	
Disposal Alternative 2: ……	
Disposal Alternative 3: ……	
General Comments:	Acid digestates containing low levels of metals can be neutralized and disposed into the sewer. However, it is preferable that the entire metals digestate waste be managed for off-site disposal.

Waste Management Form

ITEM	DESCRIPTION
Item Number	W24
Wastestream and Source	Mercury containing acid waste from samples and/or TKN/COD test procedures. Mercury from broken thermometers.
Hazardous Characteristics	Hazardous (corrosive and toxic).
On-Site Management Options:	
Sewarable? ……...	……No
Sanitary Landfillable? …….	……No
Other: ……..	……Accumulate for off-site disposal
Off-Site Management Options:	Off-site disposal
Transportation Requirements:	
DOT shipping name: …….	……..RQ Waste Corrosive Liquid, Acidic, Inorganic
Hazard Class: ……	……..
UN/NA No.: …….……	……..UN3264
EPA Waste No.: ……...	……..D002, D009
Physical State: ……..	……..Liquid
Container Type: ……	……..
Other: ……	……..
LDR information:	
Treatment Alternatives:	
Treatment Alternative 1: ……..	
Treatment Alternative 2: ……..	
Treatment Alternative 3: ……..	
Recycling Alternatives:	
Recycling Alternative 1: ……..	
Recycling Alternative 2: ……..	
Recycling Alternative 3: ……..	
Disposal Alternatives:	
Disposal Alternative 1: ……	
Disposal Alternative 2: ……	
Disposal Alternative 3: ……	
General Comments:	Accumulate small quantities within the laboratory for off-site disposal.

Waste Management Form

ITEM	DESCRIPTION
Item Number	W25
Wastestream and Source	To be Identified
Hazardous Characteristics	
On-Site Management Options:	
Sewarable? ……..	……
Sanitary Landfillable? …….	……
Other: ……..	……
Off-Site Management Options:	
Transportation Requirements:	
DOT shipping name: …….	……..
Hazard Class: ……	……..
UN/NA No.: …….……	……..
EPA Waste No.: ……...	……..
Physical State: ……..	……..
Container Type: ……	……..
Other: ……	……..
LDR information:	
Treatment Alternatives:	
Treatment Alternative 1: ……..	
Treatment Alternative 2: ……..	
Treatment Alternative 3: ……..	
Recycling Alternatives:	
Recycling Alternative 1: ……..	
Recycling Alternative 2: ……..	
Recycling Alternative 3: ……..	
Disposal Alternatives:	
Disposal Alternative 1: ……	
Disposal Alternative 2: ……	
Disposal Alternative 3: ……	
General Comments:	

STANDARD OPERATING PROCEDURES

No. Q-Water-RD

REAGENT WATER

October 31, 2005

APPROVED:

_____ _____
Operations Manager, Analytical Management Laboratories Date

_____ _____
Laboratory Director, Analytical Management Laboratories Date

_____ _____
Prepared By: Tenkasi S. Viswanathan, Ph.D. Date
Quality Assurance Director, Analytical Management Laboratories

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) provides information and guidance for the operation and maintenance of systems used to prepare reagent-grade water. It is based on Method-1080 of the Standard Methods for the Examination of Water and Wastewater, 15th edition.

2.0 SUMMARY OF METHOD

Reagent-grade water is one of the most critical but most often neglected reagent used in laboratory operations. It is used for blank analysis, LCS/LCSD analysis, sample digestion and for the dilution of other reagents. ASTM, USP, CAP and other organizations have several grades of reagent waters (Table I), which are suitable for different applications. ACS defines the reagent grade water as water that has been distilled and/or deionized so that it has a specific resistance of more than 500,000 ohms or a conductivity of 2.0 micromhos.

Reagent-grade water should be free of substances that are either included or interfere with the list of target parameters in the analytical methods. The quality of water required is related directly to the analysis being made. Failure to prepare water properly may account for the poor performance of some analytical methods.

Reagent-grade water is prepared using a combination of ion exchange, carbon adsorption, reverse osmosis, ultrafiltration, distillation, UV treatment, etc. This SOP describes the procedures that are used by Analytical Management Laboratories for the preparation, monitoring and maintenance of such systems.

3.0 INTERFERENCES

Not applicable

4.0 APPARATUS AND MATERIALS

Carbon bed
Mixed bed ion exchangers
Resistivity meter (Ionpure RM-2 in-line meter)
USFilter Purelab Classic Polishing System
Conductivity meter
Barnstead Still

5.0 REAGENTS

None

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Collect the samples in clean glass or plastic beakers for conductivity determinations.

7.0 PROCEDURE

Type II Water Preparation: Type II water is prepared by passing tap water through a carbon adsorbent bed (US Filter or equivalent source), an initial cation-anion (40:60) mixed bed equipped with a warning light, a final finishing cation-anion (40:60) mixed bed fitted with an in-line resistivity meter, and a resin filter. The warning light will change from green to red if the resistivity drops below 200,000 ohms. The warning light is monitored everyday and the first mixed bed replaced, when the light turns red. The system can be used in the interim (after replacement has been ordered) since the second finishing bed will assure quality of the water, which is identical to the first bed.

Type II Water Monitoring: Record the color (Table II) of the warning light and the resistivity (megaohm) to three significant figures every working day. Run the water for a few seconds before resistance is read on the Ionpure RM-2 resistivity meter. Contact the vendor to order replacement cartridge when the green indicator light turns red. Circle the red indicators light readings. Service to restore it to green should be completed as soon as possible, preferably within one week of its discovery.

Type I Water Preparation: Type I water is prepared by passing the Type II water through USFilter Purelab Classic water polishing system. This system has a mixed resin and carbon cartridge that removes organics and inorganics that are present in Type II water. The maximum resistance obtainable from this system is 18.2 MegaOhms.

Type I Water Monitoring: Record (Table III) the resistivity (megaohm) to three significant figures every working day. Turn on the unit and run the water for a few seconds before resistance is read on the resistivity meter. Contact the vendor to order replacement cartridge when the resistance drops below 16.7 MegaOhms. Service to restore it to green should be completed as soon as possible, preferably within one week of its discovery.

Distilled deionized water: The distilled deionized water is used for preparing trip blanks and other QC samples (e.g. MB and spiked blanks) for VOCs analysis. The still is located in the VOA lab with Type-II water as the source water. The distilled water is not used for any other purpose in the laboratory and the monitoring is based on MB analysis.

8.0 CALCULATIONS

Not applicable.

9.0 QUALITY CONTROL

Submit a sample of the Type I and Type II waters for conductivity analysis using normal laboratory procedures. This analysis should be performed quarterly during the first week of each calendar year quarter. Document the results in Table IV.

Table I
Reagent Water Specifications

Quality Parameter	Type I	Type II	Type III
Bacteria, CFU/ml	<10[a]	<1000	NS
The pH	NS	NS	5-8
Resistivity, Megaohm-cm at 25°C	>10	>1	0.1
Conductivity, micromho/cm	<0.1	<1	10
SiO_2, mg/L	<0.05	<0.1	<1
Particulate Matter[b]	0.22 micron filter	NS	NS
Organic contaminants[b]	Activated carbon[c]	NS	NS

[a] Preferably bacteria-free.
[b] Process specification; not measured by the end-user.
[c] Pretreatment, and possibly post-treatment for certain uses.
NS = not specified.

Table II
Type II Reagent Water Resistivity Monitoring Record (2 pages)

Analytical Management Laboratories
Type II Water Resistivity Record

Month:	January			February			March			April			May			June		
	Reading Light/Res.		Initials	Reading Light/Res.		Initials	Reading Light/Res.		Initials	Reading Light/Res.		Initials	Reading Light/Res.		Initials	Reading Light/Res.		Initials
Day	G/R	MΩ		G/R	MΩ		G/R	MΩ		G/R	MΩ		G/R	MΩ		G/R	MΩ	
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Supervisor Review																		

Analytical Management Laboratories
Type II Water Resistivity Record

Month:	July			August			September			October			November			December		
	Reading Light/Res.		Initials	Reading Light/Res.		Initials	Reading Light/Res.		Initials	Reading Light/Res.		Initials	Reading Light/Res.		Initials	Reading Light/Res.		Initials
Day	G/R	MΩ		G/R	MΩ		G/R	MΩ		G/R	MΩ		G/R	MΩ		G/R	MΩ	
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Supervisor Review																		

Table III
Type I Reagent Water Resistivity Monitoring Record (2 pages)

Analytical Management Laboratories
Type I Water Resistivity Record

Month:	January		February		March		April		May		June	
	Resistance MOhms	Initi als	Resistance MOhms	Initi als	Resistance Mohms	Initia ls	Resistance MOhms	Initi als	Resistance MOhms	Initi als	Resistance MOhms	Initials
Day												
1												
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30												
31												
Supervisor Review												

Analytical Management Laboratories
Type I Water Resistivity Record

Month:	July		August		September		October		November		December	
	Resistance MOhms	Initi als	Resistance MOhms	Initi als	Resistance MOhms	Initi als	Resistance MOhms	Initi als	Resistance MOhms	Initi als	Resistance MOhms	Initials
Day												
1												
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Supervisor Review												

Table IV
Type I and Type II Reagent Water Quarterly QA Check

Date	Type I Reagent Water		Type II Reagent Water	
	Instrument Reading MegaOhms	Laboratory Result MegaOhms	Instrument Reading MegaOhms	Laboratory Result MegaOhms

AML Certifications

U.S. Small Business Administration
Washington, D.C. 20416

February 5, 1999

CERTIFIED MAIL - RETURN RECEIPT REQUESTED

Bill Said, President
Analytical Management Laboratories, Inc.
15130 South Keeler
Olate, KS 66062

Dear Mr. Said:

Congratulations! Your firm has been certified as a Participant in the U.S. Small Business Administration's (SBA) 8(a) (BD) program. Your nine (9) year program term begins on the date of this letter.

Additionally, your firm has been certified as a Small Disadvantaged Business (SDB) in the Federal Government's SDB program. Your term of participation in the SDB program is concurrent with your 8(a) BD certification.

During participation in the 8(a) program, you will receive business development assistance from an assigned Business Opportunity Specialist in the Kansas City District Office located at 323 West 8th Street, Suite 501, Kansas City, Missouri 64105. The phone number is 816/374-6708. The District Office will also be able to provide you with information on the SDB program and its benefits.

Your firm will become eligible to receive 8(a) contracts after you submit a business plan using SBA Form 1010C and receive SBA's approval of the plan. We are sending a copy of this certification letter to the SBA Kansas City District Office. That office will send you the business plan form.

SBA requires that the President or Chief Executive Officer sign the Participation Agreement to show that he or she understands the conditions of 8(a) BD program participation. Please read the Agreement, sign and date and return it to the SBA Kansas City District Office at the address shown above. The second copy is for you.

Even though your firm's approved Primary Standard Industrial Classification (SIC) Code is 8734, your firm may be awarded contracts under other SIC Codes, as long as the firm is qualified to perform. In this regard, please note that contracts awarded under 8(a) BD program authority generally result from the self-marketing efforts of the 8(a) firm. While your firm's acceptance into the 8(a) BD program is not a guarantee of contract support, SBA will make every effort to provide you with assistance in your marketing efforts.

We welcome you as an 8(a) program participant and look forward to working with you.

Sincerely,

Deloride P. Ford
Acting Associate Administrator for
8(a) Business Development

Enclosures

FLORIDA DEPARTMENT OF HEALTH

State of Florida
Department of Health, Bureau of Laboratories

This is to certify that
E87892
Analytical Management Laboratories, Inc.
15130 S. Keeler Rd.
Olathe, KS 66062

has complied with Florida Administrative Code 64E-1 for the examination of Environmental samples in the following categories:

DRINKING WATER - Group II Unregulated Contaminants, Group III Unregulated Contaminants, Other Regulated Contaminants, Primary Inorganic Contaminants, Secondary Inorganic Contaminants, Synthetic Organic Contaminants

NON-POTABLE WATER - Extractable Organics, General Chemistry, Metals, Pesticides-Herbicides-PCB's, Volatile Organics

SOLID and CHEMICAL MATERIALS - Extractable Organics, General Chemistry, Metals, Pesticides-Herbicides PCB's, Volatile Organics

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

EFFECTIVE JULY 1, 2005 THROUGH JUNE 30, 2006

Ming S. Chan, Ph.D.
Bureau Chief, Bureau of Laboratories
Florida Department of Health
DH Form 1697, 7/04
NON-TRANSFERABLE 05/10/2005-E87892

Laboratory Scope of Accreditation

THIS LISTING OF ACCREDITED ANALYTES SHOULD BE USED ONLY WHEN ASSOCIATED WITH A VALID CERTIFICATE

State Laboratory ID: E87892 EPA Lab Code: KS00902 (913) 829-0101

E87892
Analytical Management Laboratories, Inc.
15130 S. Keeler Rd.
Olathe, KS 66062

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
1,1,1-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
1,1,2,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
1,1,2-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
1,1-Dichloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
1,1-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
1,1-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
1,2,3-Trichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
1,2,3-Trichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
1,2,4-Trichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
1,2,4-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
1,2-Dibromo-3-chloropropane (DBCP)	EPA 504.1	Synthetic Organic Contaminants	NELAP	2/13/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 504.1	Synthetic Organic Contaminants	NELAP	2/13/2003
1,2-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
1,2-Dichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
1,2-Dichloropropane	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
1,3,5-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
1,3-Dichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
1,3-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
1,4-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
2,2-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 525.2	Group III Unregulated Contaminants	NELAP	2/13/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 525.2	Group III Unregulated Contaminants	NELAP	2/13/2003
2-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
4-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
4-Isopropyltoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
Aluminum	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/13/2003
Aluminum	EPA 200.8	Secondary Inorganic Contaminants	NELAP	2/13/2003
Antimony	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/13/2003
Arsenic	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/13/2003
Arsenic	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/13/2003
Barium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/13/2003
Barium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/13/2003
Benzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
Benzo(a)pyrene	EPA 525.2	Synthetic Organic Contaminants	NELAP	2/13/2003
Beryllium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/13/2003

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State Laboratory ID: E87892 **EPA Lab Code:** KS00902 **(913) 829-0101**

E87892
Analytical Management Laboratories, Inc.
15130 S. Keeler Rd.
Olathe, KS 66062

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Beryllium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/13/2003
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 525.2	Synthetic Organic Contaminants	NELAP	2/13/2003
Bromobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
Bromochloromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
Bromodichloromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
Cadmium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/13/2003
Cadmium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/13/2003
Calcium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/13/2003
Carbon tetrachloride	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
Chlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
Chloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
Chloroform	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
Chromium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/13/2003
Chromium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/13/2003
cis-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
cis-1,3-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
Copper	EPA 200.7	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	2/13/2003
Copper	EPA 200.8	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	2/13/2003
Di(2-ethylhexyl)adipate	EPA 525.2	Synthetic Organic Contaminants	NELAP	2/13/2003
Dibromochloromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
Dibromomethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
Dichlorodifluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
Dichloromethane (DCM, Methylene chloride)	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
Ethylbenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
Iron	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/13/2003
Isopropylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
Lead	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/13/2003
Manganese	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/13/2003
Manganese	EPA 200.8	Secondary Inorganic Contaminants	NELAP	2/13/2003
Mercury	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/13/2003
Mercury	EPA 245.1	Primary Inorganic Contaminants	NELAP	2/13/2003
Mercury	EPA 245.2	Primary Inorganic Contaminants	NELAP	2/13/2003
Methyl bromide (Bromomethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
Methyl chloride (Chloromethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003

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State Laboratory ID: E87892 **EPA Lab Code:** KS00902 (913) 829-0101

E87892
Analytical Management Laboratories, Inc.
15130 S. Keeler Rd.
Olathe, KS 66062

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Methyl tert-butyl ether (MTBE)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
Naphthalene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
n-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
Nickel	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/13/2003
Nickel	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/13/2003
Nitrobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
n-Propylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
sec-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
Selenium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/13/2003
Sodium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/13/2003
Styrene	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
tert-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
Tetrachloroethylene (Perchloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
Thallium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/13/2003
Toluene	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
trans-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
trans-1,3-Dichloropropylene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/13/2003
Trichloroethene (Trichloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
Vinyl chloride	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
Xylene (total)	EPA 524.2	Other Regulated Contaminants	NELAP	2/13/2003
Zinc	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/13/2003
Zinc	EPA 200.8	Secondary Inorganic Contaminants	NELAP	2/13/2003

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State Laboratory ID: E87892 **EPA Lab Code:** KS00902 (913) 829-0101

E87892
Analytical Management Laboratories, Inc.
15130 S. Keeler Rd.
Olathe, KS 66062

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1-Trichloroethane	EPA 624	Volatile Organics	NELAP	2/13/2003
1,1,2,2-Tetrachloroethane	EPA 624	Volatile Organics	NELAP	2/13/2003
1,1,2-Trichloroethane	EPA 624	Volatile Organics	NELAP	2/13/2003
1,1-Dichloroethane	EPA 624	Volatile Organics	NELAP	2/13/2003
1,1-Dichloroethylene	EPA 624	Volatile Organics	NELAP	2/13/2003
1,2,4-Trichlorobenzene	EPA 625	Extractable Organics	NELAP	2/13/2003
1,2-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	2/13/2003
1,2-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	2/13/2003
1,2-Dichloroethane	EPA 624	Volatile Organics	NELAP	2/13/2003
1,2-Dichloropropane	EPA 624	Volatile Organics	NELAP	2/13/2003
1,3-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	2/13/2003
1,3-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	2/13/2003
1,4-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	2/13/2003
1,4-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	2/13/2003
2,4,6-Trichlorophenol	EPA 625	Extractable Organics	NELAP	2/13/2003
2,4-Dichlorophenol	EPA 625	Extractable Organics	NELAP	2/13/2003
2,4-Dimethylphenol	EPA 625	Extractable Organics	NELAP	2/13/2003
2,4-Dinitrophenol	EPA 625	Extractable Organics	NELAP	2/13/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 625	Extractable Organics	NELAP	2/13/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 625	Extractable Organics	NELAP	2/13/2003
2-Chloroethyl vinyl ether	EPA 624	Volatile Organics	NELAP	2/13/2003
2-Chloronaphthalene	EPA 625	Extractable Organics	NELAP	2/13/2003
2-Chlorophenol	EPA 625	Extractable Organics	NELAP	2/13/2003
2-Methyl-4,6-dinitrophenol	EPA 625	Extractable Organics	NELAP	2/13/2003
2-Nitrophenol	EPA 625	Extractable Organics	NELAP	2/13/2003
3,3'-Dichlorobenzidine	EPA 625	Extractable Organics	NELAP	2/13/2003
4,4'-DDD	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
4,4'-DDE	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
4,4'-DDT	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
4-Bromophenyl phenyl ether	EPA 625	Extractable Organics	NELAP	2/13/2003
4-Chloro-3-methylphenol	EPA 625	Extractable Organics	NELAP	2/13/2003
4-Chlorophenyl phenylether	EPA 625	Extractable Organics	NELAP	2/13/2003
4-Nitrophenol	EPA 625	Extractable Organics	NELAP	2/13/2003
Acenaphthene	EPA 625	Extractable Organics	NELAP	2/13/2003
Acenaphthylene	EPA 625	Extractable Organics	NELAP	2/13/2003
Acidity, as CaCO3	EPA 305.1	General Chemistry	NELAP	2/13/2003

THIS LISTING OF ACCREDITED ANALYTES SHOULD BE USED ONLY WHEN ASSOCIATED WITH A VALID CERTIFICATE

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E87892
Analytical Management Laboratories, Inc.
15130 S. Keeler Rd.
Olathe, KS 66062

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Acrolein (Propenal)	EPA 624	Volatile Organics	NELAP	2/13/2003
Acrylonitrile	EPA 624	Volatile Organics	NELAP	2/13/2003
Aldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Alkalinity as CaCO3	EPA 310.1	General Chemistry	NELAP	2/13/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Aluminum	EPA 200.7	Metals	NELAP	2/13/2003
Aluminum	EPA 200.8	Metals	NELAP	2/13/2003
Ammonia as N	EPA 350.2	General Chemistry	NELAP	2/13/2003
Anthracene	EPA 625	Extractable Organics	NELAP	2/13/2003
Antimony	EPA 200.7	Metals	NELAP	2/13/2003
Antimony	EPA 200.8	Metals	NELAP	2/13/2003
Antimony	EPA 200.9	Metals	NELAP	2/13/2003
Aroclor-1016 (PCB-1016)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Aroclor-1221 (PCB-1221)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Aroclor-1232 (PCB-1232)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Aroclor-1242 (PCB-1242)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Aroclor-1248 (PCB-1248)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Aroclor-1254 (PCB-1254)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Aroclor-1260 (PCB-1260)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Arsenic	EPA 200.7	Metals	NELAP	2/13/2003
Arsenic	EPA 200.8	Metals	NELAP	2/13/2003
Arsenic	EPA 200.9	Metals	NELAP	2/13/2003
Arsenic	EPA 6010	Metals	NELAP	2/13/2003
Arsenic	EPA 6020	Metals	NELAP	2/13/2003
Arsenic	EPA 7060	Metals	NELAP	2/13/2003
Barium	EPA 200.7	Metals	NELAP	2/13/2003
Barium	EPA 200.8	Metals	NELAP	2/13/2003
Benzene	EPA 624	Volatile Organics	NELAP	2/13/2003
Benzidine	EPA 625	Extractable Organics	NELAP	2/13/2003
Benzo(a)anthracene	EPA 625	Extractable Organics	NELAP	2/13/2003
Benzo(a)pyrene	EPA 625	Extractable Organics	NELAP	2/13/2003
Benzo(b)fluoranthene	EPA 625	Extractable Organics	NELAP	2/13/2003
Benzo(g,h,i)perylene	EPA 625	Extractable Organics	NELAP	2/13/2003
Benzo(k)fluoranthene	EPA 625	Extractable Organics	NELAP	2/13/2003
Beryllium	EPA 200.7	Metals	NELAP	2/13/2003
Beryllium	EPA 200.8	Metals	NELAP	2/13/2003

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State Laboratory ID: E87892 **EPA Lab Code:** KS00902 (913) 829-0101

E87892
Analytical Management Laboratories, Inc.
15130 S. Keeler Rd.
Olathe, KS 66062

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
beta-BHC (beta-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Biochemical oxygen demand	EPA 405.1	General Chemistry	NELAP	2/13/2003
Biochemical oxygen demand	SM 5210 B	General Chemistry	NELAP	2/13/2003
bis(2-Chloroethoxy)methane	EPA 625	Extractable Organics	NELAP	2/13/2003
bis(2-Chloroethyl) ether	EPA 625	Extractable Organics	NELAP	2/13/2003
bis(2-Chloroisopropyl) ether	EPA 625	Extractable Organics	NELAP	2/13/2003
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 625	Extractable Organics	NELAP	2/13/2003
Bromide	EPA 300.0	General Chemistry	NELAP	2/13/2003
Bromodichloromethane	EPA 624	Volatile Organics	NELAP	2/13/2003
Bromoform	EPA 624	Volatile Organics	NELAP	2/13/2003
Butyl benzyl phthalate	EPA 625	Extractable Organics	NELAP	2/13/2003
Cadmium	EPA 200.7	Metals	NELAP	2/13/2003
Cadmium	EPA 200.8	Metals	NELAP	2/13/2003
Cadmium	EPA 6010	Metals	NELAP	2/13/2003
Cadmium	EPA 6020	Metals	NELAP	2/13/2003
Calcium	EPA 200.7	Metals	NELAP	2/13/2003
Carbon tetrachloride	EPA 624	Volatile Organics	NELAP	2/13/2003
Chemical oxygen demand	EPA 410.4	General Chemistry	NELAP	2/13/2003
Chemical oxygen demand	OTHER HACH 8000	General Chemistry	NELAP	2/13/2003
Chlordane (tech.)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Chloride	EPA 300.0	General Chemistry	NELAP	2/13/2003
Chloride	EPA 325.3	General Chemistry	NELAP	2/13/2003
Chlorobenzene	EPA 624	Volatile Organics	NELAP	2/13/2003
Chloroethane	EPA 624	Volatile Organics	NELAP	2/13/2003
Chloroform	EPA 624	Volatile Organics	NELAP	2/13/2003
Chromium	EPA 200.7	Metals	NELAP	2/13/2003
Chromium	EPA 200.8	Metals	NELAP	2/13/2003
Chromium	EPA 200.9	Metals	NELAP	2/13/2003
Chromium	EPA 6010	Metals	NELAP	2/13/2003
Chromium	EPA 6020	Metals	NELAP	2/13/2003
Chrysene	EPA 625	Extractable Organics	NELAP	2/13/2003
cis-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	2/13/2003
Cobalt	EPA 200.7	Metals	NELAP	2/13/2003
Cobalt	EPA 200.8	Metals	NELAP	2/13/2003
Color	EPA 110.3	General Chemistry	NELAP	2/13/2003
Conductivity	EPA 120.1	General Chemistry	NELAP	2/13/2003

Laboratory Scope of Accreditation

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State Laboratory ID: E87892 EPA Lab Code: KS00902 (913) 829-0101

E87892
Analytical Management Laboratories, Inc.
15130 S. Keeler Rd.
Olathe, KS 66062

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Copper	EPA 200.7	Metals	NELAP	2/13/2003
Copper	EPA 200.8	Metals	NELAP	2/13/2003
Copper	EPA 6010	Metals	NELAP	2/13/2003
Copper	EPA 6020	Metals	NELAP	2/13/2003
delta-BHC	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Dibenz(a,h) anthracene	EPA 625	Extractable Organics	NELAP	2/13/2003
Dibromochloromethane	EPA 624	Volatile Organics	NELAP	2/13/2003
Dieldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Diethyl phthalate	EPA 625	Extractable Organics	NELAP	2/13/2003
Dimethyl phthalate	EPA 625	Extractable Organics	NELAP	2/13/2003
Di-n-butyl phthalate	EPA 625	Extractable Organics	NELAP	2/13/2003
Di-n-octyl phthalate	EPA 625	Extractable Organics	NELAP	2/13/2003
Endosulfan I	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Endosulfan II	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Endosulfan sulfate	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Endrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Endrin aldehyde	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Ethylbenzene	EPA 624	Volatile Organics	NELAP	2/13/2003
Fluoranthene	EPA 625	Extractable Organics	NELAP	2/13/2003
Fluorene	EPA 625	Extractable Organics	NELAP	2/13/2003
Fluoride	EPA 300.0	General Chemistry	NELAP	2/13/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Hardness	SM 2340 B	General Chemistry	NELAP	2/13/2003
Heptachlor	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Heptachlor epoxide	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Hexachlorobenzene	EPA 625	Extractable Organics	NELAP	2/13/2003
Hexachlorobutadiene	EPA 625	Extractable Organics	NELAP	2/13/2003
Hexachlorocyclopentadiene	EPA 625	Extractable Organics	NELAP	2/13/2003
Hexachloroethane	EPA 625	Extractable Organics	NELAP	2/13/2003
Indeno(1,2,3-cd)pyrene	EPA 625	Extractable Organics	NELAP	2/13/2003
Iron	EPA 200.7	Metals	NELAP	2/13/2003
Isophorone	EPA 625	Extractable Organics	NELAP	2/13/2003
Lead	EPA 200.7	Metals	NELAP	2/13/2003
Lead	EPA 200.8	Metals	NELAP	2/13/2003
Lead	EPA 200.9	Metals	NELAP	2/13/2003
Lead	EPA 6010	Metals	NELAP	2/13/2003

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E87892
Analytical Management Laboratories, Inc.
15130 S. Keeler Rd.
Olathe, KS 66062

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Lead	EPA 6020	Metals	NELAP	2/13/2003
Lead	EPA 7421	Metals	NELAP	2/13/2003
Magnesium	EPA 200.7	Metals	NELAP	2/13/2003
Manganese	EPA 200.7	Metals	NELAP	2/13/2003
Manganese	EPA 200.8	Metals	NELAP	2/13/2003
Mercury	EPA 200.8	Metals	NELAP	2/13/2003
Mercury	EPA 245.1	Metals	NELAP	2/13/2003
Mercury	EPA 245.2	Metals	NELAP	2/13/2003
Mercury	EPA 7470	Metals	NELAP	2/13/2003
Mercury	EPA 7471	Metals	NELAP	2/13/2003
Methyl bromide (Bromomethane)	EPA 624	Volatile Organics	NELAP	2/13/2003
Methyl chloride (Chloromethane)	EPA 624	Volatile Organics	NELAP	2/13/2003
Methylene chloride	EPA 624	Volatile Organics	NELAP	2/13/2003
Molybdenum	EPA 200.7	Metals	NELAP	2/13/2003
Molybdenum	EPA 200.8	Metals	NELAP	2/13/2003
Molybdenum	EPA 6010	Metals	NELAP	2/13/2003
Molybdenum	EPA 6020	Metals	NELAP	2/13/2003
Naphthalene	EPA 625	Extractable Organics	NELAP	2/13/2003
Nickel	EPA 200.7	Metals	NELAP	2/13/2003
Nickel	EPA 200.8	Metals	NELAP	2/13/2003
Nickel	EPA 6010	Metals	NELAP	2/13/2003
Nickel	EPA 6020	Metals	NELAP	2/13/2003
Nitrate as N	EPA 300.0	General Chemistry	NELAP	2/13/2003
Nitrate as N	EPA 353.3	General Chemistry	NELAP	2/13/2003
Nitrite as N	EPA 354.1	General Chemistry	NELAP	2/13/2003
Nitrobenzene	EPA 625	Extractable Organics	NELAP	2/13/2003
n-Nitrosodimethylamine	EPA 625	Extractable Organics	NELAP	2/13/2003
n-Nitrosodi-n-propylamine	EPA 625	Extractable Organics	NELAP	2/13/2003
n-Nitrosodiphenylamine	EPA 625	Extractable Organics	NELAP	2/13/2003
Organic nitrogen	TKN minus AMMONIA	General Chemistry	NELAP	2/13/2003
Orthophosphate as P	EPA 300.0	General Chemistry	NELAP	2/13/2003
Orthophosphate as P	EPA 365.3	General Chemistry	NELAP	2/13/2003
Oxygen, dissolved	EPA 360.1	General Chemistry	NELAP	2/13/2003
Pentachlorophenol	EPA 625	Extractable Organics	NELAP	2/13/2003
pH	EPA 150.1	General Chemistry	NELAP	2/13/2003
Phenanthrene	EPA 625	Extractable Organics	NELAP	2/13/2003

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State Laboratory ID: E87892 **EPA Lab Code:** KS00902 **(913) 829-0101**

E87892
Analytical Management Laboratories, Inc.
15130 S. Keeler Rd.
Olathe, KS 66062

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Phenol	EPA 625	Extractable Organics	NELAP	2/13/2003
Potassium	EPA 200.7	Metals	NELAP	2/13/2003
Pyrene	EPA 625	Extractable Organics	NELAP	2/13/2003
Residue-filterable (TDS)	EPA 160.1	General Chemistry	NELAP	2/13/2003
Residue-nonfilterable (TSS)	EPA 160.2	General Chemistry	NELAP	2/13/2003
Residue-settleable	EPA 160.5	General Chemistry	NELAP	2/13/2003
Residue-total	EPA 160.3	General Chemistry	NELAP	2/13/2003
Residue-volatile	EPA 160.4	General Chemistry	NELAP	2/13/2003
Selenium	EPA 200.7	Metals	NELAP	2/13/2003
Selenium	EPA 200.8	Metals	NELAP	2/13/2003
Selenium	EPA 200.9	Metals	NELAP	2/13/2003
Selenium	EPA 6010	Metals	NELAP	2/13/2003
Silver	EPA 200.7	Metals	NELAP	2/13/2003
Silver	EPA 200.8	Metals	NELAP	2/13/2003
Sodium	EPA 200.7	Metals	NELAP	2/13/2003
Sulfate	EPA 300.0	General Chemistry	NELAP	2/13/2003
Sulfate	EPA 375.4	General Chemistry	NELAP	2/13/2003
Sulfide	EPA 376.2	General Chemistry	NELAP	2/13/2003
Temperature, deg. C	EPA 170.1	General Chemistry	NELAP	2/13/2003
Tetrachloroethylene (Perchloroethylene)	EPA 624	Volatile Organics	NELAP	2/13/2003
Thallium	EPA 200.7	Metals	NELAP	2/13/2003
Thallium	EPA 200.8	Metals	NELAP	2/13/2003
Thallium	EPA 200.9	Metals	NELAP	2/13/2003
Toluene	EPA 624	Volatile Organics	NELAP	2/13/2003
Total nitrate-nitrite	EPA 353.3	General Chemistry	NELAP	2/13/2003
Total organic carbon	SM 5310B	General Chemistry	NELAP	2/13/2003
Total residual chlorine	EPA 330.4	General Chemistry	NELAP	2/13/2003
Toxaphene (Chlorinated camphene)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
trans-1,2-Dichloroethylene	EPA 624	Volatile Organics	NELAP	2/13/2003
trans-1,3-Dichloropropylene	EPA 624	Volatile Organics	NELAP	2/13/2003
Trichloroethene (Trichloroethylene)	EPA 624	Volatile Organics	NELAP	2/13/2003
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	2/13/2003
Vanadium	EPA 200.7	Metals	NELAP	2/13/2003
Vanadium	EPA 200.8	Metals	NELAP	2/13/2003
Vinyl chloride	EPA 624	Volatile Organics	NELAP	2/13/2003
Zinc	EPA 200.7	Metals	NELAP	2/13/2003

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State Laboratory ID: E87892 EPA Lab Code: KS00902 (913) 829-0101

E87892
Analytical Management Laboratories, Inc.
15130 S. Keeler Rd.
Olathe, KS 66062

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Zinc	EPA 200.8	Metals	NELAP	2/13/2003
Zinc	EPA 6010	Metals	NELAP	2/13/2003
Zinc	EPA 6020	Metals	NELAP	2/13/2003

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State Laboratory ID: E87892 **EPA Lab Code:** KS00902 (913) 829-0101

E87892
Analytical Management Laboratories, Inc.
15130 S. Keeler Rd.
Olathe, KS 66062

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	2/13/2003
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/13/2003
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/13/2003
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	2/13/2003
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	2/13/2003
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/13/2003
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	2/13/2003
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/13/2003
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/13/2003
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/13/2003
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	2/13/2003
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	2/13/2003
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	2/13/2003
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	2/13/2003
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	2/13/2003
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	2/13/2003

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E87892
Analytical Management Laboratories, Inc.
15130 S. Keeler Rd.
Olathe, KS 66062

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	2/13/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	2/13/2003
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	2/13/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	2/13/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	2/13/2003
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	2/13/2003
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	2/13/2003
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	2/13/2003
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	2/13/2003
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	2/13/2003
2-Hexanone	EPA 8260	Volatile Organics	NELAP	2/13/2003
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	2/13/2003
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	2/13/2003
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	2/13/2003
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	2/13/2003
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	2/13/2003
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	2/13/2003
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	2/13/2003
3-Methylphenol (m-Cresol)	EPA 8270	Extractable Organics	NELAP	2/13/2003
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	2/13/2003
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	2/13/2003
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	2/13/2003
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	2/13/2003
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	2/13/2003
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	2/13/2003
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	2/13/2003
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	2/13/2003
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	2/13/2003
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	2/13/2003
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	2/13/2003
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	2/13/2003
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	2/13/2003
Acenaphthene	EPA 8270	Extractable Organics	NELAP	2/13/2003

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Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Acenaphthene	EPA 8310	Extractable Organics	NELAP	2/13/2003
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Acenaphthylene	EPA 8310	Extractable Organics	NELAP	2/13/2003
Acetone	EPA 8260	Volatile Organics	NELAP	2/13/2003
Acetonitrile	EPA 8260	Volatile Organics	NELAP	2/13/2003
Acetophenone	EPA 8270	Extractable Organics	NELAP	2/13/2003
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	2/13/2003
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	2/13/2003
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Aluminum	EPA 6010	Metals	NELAP	2/13/2003
Aluminum	EPA 6020	Metals	NELAP	2/13/2003
Amenable cyanide	EPA 9010/9014	General Chemistry	NELAP	2/13/2003
Aniline	EPA 8270	Extractable Organics	NELAP	2/13/2003
Anthracene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Anthracene	EPA 8310	Extractable Organics	NELAP	2/13/2003
Antimony	EPA 6010	Metals	NELAP	2/13/2003
Antimony	EPA 6020	Metals	NELAP	2/13/2003
Antimony	EPA 7041	Metals	NELAP	2/13/2003
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Arsenic	EPA 6010	Metals	NELAP	2/13/2003
Arsenic	EPA 6020	Metals	NELAP	2/13/2003
Arsenic	EPA 7060	Metals	NELAP	2/13/2003
Barium	EPA 6010	Metals	NELAP	2/13/2003
Barium	EPA 6020	Metals	NELAP	2/13/2003
Benzene	EPA 8260	Volatile Organics	NELAP	2/13/2003
Benzidine	EPA 8270	Extractable Organics	NELAP	2/13/2003
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Benzo(a)anthracene	EPA 8310	Extractable Organics	NELAP	2/13/2003

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State Laboratory ID: E87892 EPA Lab Code: KS00902 (913) 829-0101

E87892
Analytical Management Laboratories, Inc.
15130 S. Keeler Rd.
Olathe, KS 66062

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Benzo(a)pyrene	EPA 8310	Extractable Organics	NELAP	2/13/2003
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Benzo(b)fluoranthene	EPA 8310	Extractable Organics	NELAP	2/13/2003
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Benzo(g,h,i)perylene	EPA 8310	Extractable Organics	NELAP	2/13/2003
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Benzo(k)fluoranthene	EPA 8310	Extractable Organics	NELAP	2/13/2003
Benzoic acid	EPA 8270	Extractable Organics	NELAP	2/13/2003
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	2/13/2003
Beryllium	EPA 6010	Metals	NELAP	2/13/2003
Beryllium	EPA 6020	Metals	NELAP	2/13/2003
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
beta-Naphthylamine	EPA 8270	Extractable Organics	NELAP	2/13/2003
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	2/13/2003
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	2/13/2003
bis(2-Chloroisopropyl) ether	EPA 8270	Extractable Organics	NELAP	2/13/2003
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	2/13/2003
Bromide	EPA 9056	General Chemistry	NELAP	2/13/2003
Bromobenzene	EPA 8260	Volatile Organics	NELAP	2/13/2003
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	2/13/2003
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	2/13/2003
Bromoform	EPA 8260	Volatile Organics	NELAP	2/13/2003
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	2/13/2003
Cadmium	EPA 6010	Metals	NELAP	2/13/2003
Cadmium	EPA 6020	Metals	NELAP	2/13/2003
Calcium	EPA 6010	Metals	NELAP	2/13/2003
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	2/13/2003
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	2/13/2003
Cation exchange capacity	EPA 9080	General Chemistry	NELAP	2/13/2003
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Chloride	EPA 9056	General Chemistry	NELAP	2/13/2003
Chlorine	EPA 9077	General Chemistry	NELAP	2/13/2003
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	2/13/2003
Chloroethane	EPA 8260	Volatile Organics	NELAP	2/13/2003
Chloroform	EPA 8260	Volatile Organics	NELAP	2/13/2003

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State Laboratory ID: E87892 **EPA Lab Code:** KS00902 **(913) 829-0101**

E87892
Analytical Management Laboratories, Inc.
15130 S. Keeler Rd.
Olathe, KS 66062

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Chromium	EPA 6010	Metals	NELAP	2/13/2003
Chromium	EPA 6020	Metals	NELAP	2/13/2003
Chromium	EPA 7191	Metals	NELAP	2/13/2003
Chromium VI	EPA 7195	Metals	NELAP	2/13/2003
Chromium VI	EPA 7196	General Chemistry	NELAP	2/13/2003
Chrysene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Chrysene	EPA 8310	Extractable Organics	NELAP	2/13/2003
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/13/2003
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	2/13/2003
Cobalt	EPA 6010	Metals	NELAP	2/13/2003
Cobalt	EPA 6020	Metals	NELAP	2/13/2003
Conductivity	EPA 9050	General Chemistry	NELAP	2/13/2003
Copper	EPA 6010	Metals	NELAP	2/13/2003
Copper	EPA 6020	Metals	NELAP	2/13/2003
Corrosivity	EPA 1110	General Chemistry	NELAP	2/13/2003
Dacthal (DCPA)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Dibenz(a,h) anthracene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Dibenz(a,h) anthracene	EPA 8310	Extractable Organics	NELAP	2/13/2003
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	2/13/2003
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	2/13/2003
Dibromomethane	EPA 8260	Volatile Organics	NELAP	2/13/2003
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	2/13/2003
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	2/13/2003
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	2/13/2003
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	2/13/2003
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	2/13/2003
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Diphenylamine	EPA 8270	Extractable Organics	NELAP	2/13/2003
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003

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E87892
Analytical Management Laboratories, Inc.
15130 S. Keeler Rd.
Olathe, KS 66062

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	2/13/2003
Fluoranthene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Fluoranthene	EPA 8310	Extractable Organics	NELAP	2/13/2003
Fluorene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Fluorene	EPA 8310	Extractable Organics	NELAP	2/13/2003
Fluoride	EPA 9056	General Chemistry	NELAP	2/13/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	2/13/2003
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	2/13/2003
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Ignitability	EPA 1010	General Chemistry	NELAP	2/13/2003
Ignitability	EPA 1020	General Chemistry	NELAP	2/13/2003
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Indeno(1,2,3-cd)pyrene	EPA 8310	Extractable Organics	NELAP	2/13/2003
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	2/13/2003
Iron	EPA 6010	Metals	NELAP	2/13/2003
Isophorone	EPA 8270	Extractable Organics	NELAP	2/13/2003
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	2/13/2003
Lead	EPA 6010	Metals	NELAP	2/13/2003
Lead	EPA 6020	Metals	NELAP	2/13/2003
Lead	EPA 7421	Metals	NELAP	2/13/2003
Magnesium	EPA 6010	Metals	NELAP	2/13/2003
Manganese	EPA 6010	Metals	NELAP	2/13/2003
Manganese	EPA 6020	Metals	NELAP	2/13/2003
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Mercury	EPA 7470	Metals	NELAP	2/13/2003
Mercury	EPA 7471	Metals	NELAP	2/13/2003
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003

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E87892
Analytical Management Laboratories, Inc.
15130 S. Keeler Rd.
Olathe, KS 66062

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	2/13/2003
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	2/13/2003
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	2/13/2003
Methylene chloride	EPA 8260	Volatile Organics	NELAP	2/13/2003
Molybdenum	EPA 6010	Metals	NELAP	2/13/2003
Naphthalene	EPA 8260	Volatile Organics	NELAP	2/13/2003
Naphthalene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Naphthalene	EPA 8310	Extractable Organics	NELAP	2/13/2003
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/13/2003
Nickel	EPA 6010	Metals	NELAP	2/13/2003
Nickel	EPA 6020	Metals	NELAP	2/13/2003
Nitrate	EPA 9056	General Chemistry	NELAP	2/13/2003
Nitrite	EPA 9056	General Chemistry	NELAP	2/13/2003
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	2/13/2003
Nitroglycerin	EPA 8332	Extractable Organics	NELAP	2/13/2003
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	2/13/2003
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	2/13/2003
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	2/13/2003
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	2/13/2003
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	2/13/2003
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	2/13/2003
Oil & Grease	EPA 9070	General Chemistry	NELAP	2/13/2003
Orthophosphate as P	EPA 9056	General Chemistry	NELAP	2/13/2003
Paint Filter Liquids Test	EPA 9095	General Chemistry	NELAP	2/13/2003
Pentachlorophenol	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	2/13/2003
pH	EPA 9040	General Chemistry	NELAP	2/13/2003
pH	EPA 9041	General Chemistry	NELAP	2/13/2003
pH	EPA 9045	General Chemistry	NELAP	2/13/2003
Phenanthrene	EPA 8270	Extractable Organics	NELAP	2/13/2003
Phenanthrene	EPA 8310	Extractable Organics	NELAP	2/13/2003
Phenol	EPA 8270	Extractable Organics	NELAP	2/13/2003
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	2/13/2003
Potassium	EPA 6010	Metals	NELAP	2/13/2003
Pyrene	EPA 8270	Extractable Organics	NELAP	2/13/2003

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15130 S. Keeler Rd.
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Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Pyrene	EPA 8310	Extractable Organics	NELAP	2/13/2003
Pyridine	EPA 8270	Extractable Organics	NELAP	2/13/2003
RDX (hexahydro-1,3,5-trinitro-1,3,5-trinzine)	EPA 8330	Extractable Organics	NELAP	2/13/2003
Reactive cyanide	Sec. 7.3 SW-846	General Chemistry	NELAP	2/13/2003
Reactive sulfide	Sec. 7.3 SW-846	General Chemistry	NELAP	2/13/2003
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/13/2003
Selenium	EPA 6010	Metals	NELAP	2/13/2003
Silver	EPA 6010	Metals	NELAP	2/13/2003
Sodium	EPA 6010	Metals	NELAP	2/13/2003
Styrene	EPA 8260	Volatile Organics	NELAP	2/13/2003
Sulfate	EPA 9038	General Chemistry	NELAP	2/13/2003
Sulfate	EPA 9056	General Chemistry	NELAP	2/13/2003
Sulfide	EPA 9030/9034	General Chemistry	NELAP	2/13/2003
Sulfide	EPA 9030/9215	General Chemistry	NELAP	2/13/2003
Synthetic Precipitation Leaching Procedure	EPA 1312	General Chemistry	NELAP	2/13/2003
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/13/2003
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	2/13/2003
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	2/13/2003
Thallium	EPA 6010	Metals	NELAP	2/13/2003
Thallium	EPA 6020	Metals	NELAP	2/13/2003
Thallium	EPA 7841	Metals	NELAP	2/13/2003
Toluene	EPA 8260	Volatile Organics	NELAP	2/13/2003
Total cyanide	EPA 9010/9014	General Chemistry	NELAP	2/13/2003
Total nitrate-nitrite	EPA 9056	General Chemistry	NELAP	2/13/2003
Total organic carbon	EPA 9060	General Chemistry	NELAP	2/13/2003
Total phenolics	EPA 9065	General Chemistry	NELAP	2/13/2003
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/13/2003
Toxicity Characteristic Leaching Procedure	EPA 1311	General Chemistry	NELAP	2/13/2003
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/13/2003
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	2/13/2003
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	2/13/2003
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	2/13/2003
Vanadium	EPA 6010	Metals	NELAP	2/13/2003
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	2/13/2003
Xylene (total)	EPA 8260	Volatile Organics	NELAP	2/13/2003
Zinc	EPA 6010	Metals	NELAP	2/13/2003

Laboratory Scope of Accreditation

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E87892
Analytical Management Laboratories, Inc.
15130 S. Keeler Rd.
Olathe, KS 66062

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Zinc	EPA 6020	Metals	NELAP	2/13/2003

DEPARTMENT OF THE NAVY

NAVAL FACILITIES ENGINEERING SERVICE CENTER
1100 23RD AVE
PORT HUENEME CA 93043-4370

IN REPLY REFER TO:

NFESC 413
March 9, 2006

Mr. Tenkasi Viswanathan
Analytical Management Laboratories
15130 South Keeler
Olathe, Kansas 66062

Dear Mr. Viswanathan,

This correspondence addresses the status of Analytical Management Laboratories of Olathe, Kansas in the Navy Installation Restoration (IR) Quality Assurance (QA) Program as administered by the Naval Facilities Engineering Service Center (NFESC).

Your laboratory is accepted to perform sample analysis for the methods listed in Table 1. The period of acceptance expires October 31, 2006. This acceptance does not guarantee the delivery of any analytical samples. Acceptance is facility specific and can not be transferred to an affiliated or subcontract laboratory.

The Navy's assessment included a review of the laboratory's QA manual, selected standard operating procedures (SOPs) and SOP master list, list of major analytical instrumentation, performance test (PT) results and onsite audit documentation[1].

The Navy reserves the right to conduct additional laboratory assessments or to suspend or revoke acceptance status for any or all of the listed parameters if deemed necessary.

Table 1

Method	Parameter	Matrix
9056/300 Series	Anions: Bromide, Chloride, Fluoride, Nitrate, Nitrite, Orthophosphate, Phosphorus, Sulfate, Sulfide, Sulfite	Water/Solid
314.0	Perchlorate	Water
8015M	Gasoline Range Organics/Diesel Range Organics	Water/Solid
8260B	Volatile Organic Compounds	Water/Solid
8270C	Semivolatile Organic Compounds/Polyaromatic Hydrocarbon	Water/Solid
8021B	Volatile Organic Compounds	Water/Solid
8081A	Organochlorine Pesticides	Water/Solid

[1] The State of Kansas Department of Health Bureau of Laboratories conducted an on-site assessment under National Environmental Laboratory Accreditation Conference (NELAC) requirements on July 27-28, 2004 and on August 5, 2004.

NFESC 413
March 9, 2006

8082	Polychlorinated Biphenyls (PCBs)	Water/Solid/Oil
8151A	Herbicides	Water/Solid
6010B/7000A/6020	TAL Metals: Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Mercury, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Thallium, Vanadium, and Zinc	Water/Solid
8330	Explosives	Water/Solid

Acceptance for use for parameters not identified on the table will be determined by Navy project personnel.

The laboratory should notify NFESC if there are parameters not presented on Table 1 that the laboratory expects to run on a routine basis in support of Navy installation restoration projects. In these circumstances the laboratory's capability to run the tests will be reviewed and the table will be modified accordingly.

Questions concerning the information provided should be directed to the NFESC IR QA Program coordinator, Ms. Patricia Moreno at (805) 982-1659, or via email at pati.moreno@navy.mil.

Sincerely,

FOR Robert J. Kratzke
Supervisor, Consultation/Information
Management Branch

Nebraska
Department of Health and Human Services
Regulation and Licensure

Certification for the analysis of private water samples has been granted to

ANALYTICAL MANAGEMENT LABORATORIES INC.

for the following

Analytes and Test Methods as cited on the State of Kansas Department of Health and Environment Certificate (copy attached)

Under the authority of the Nebraska Safe Drinking Water Act (Neb. Rev. Stat. 71-5306 et. seq.) In order to test compliance samples from Public Water Systems, your Laboratory must enter into an agreement with NHHS, Regulation and Licensure, Department of Health and Human Services.

May 1, 2005	April 30, 2006
Certification Date	*Expiration Date*
Sandra Irons	Robert Leopold
Sandra Irons *State Certification Officer*	*Robert Leopold* *Public Health Assurance, Divison Administrator*

Nebraska Health and Human Services System

STATE OF NORTH CAROLINA DEPARTMENT OF THE
ENVIRONMENT AND NATURAL RESOURCES

DIVISION OF WATER QUALITY
LABORATORY CERTIFICATION PROGRAM

In accordance with the provisions of N.C.G.S. 143-215.3 (a) (1), 143-215.3 (a)(10) and NCAC 2H.0800:

ANALYTICAL MANAGEMENT LABS, INC.

Is hereby certified to perform environmental analysis as listed on Attachment I and report monitoring data to DWQ for compliance with NPDES effluent, surface water, groundwater, and pretreatment regulations.

By reference 15A NCAC 2H .0800 is made a part of this certificate.

This certificate does not guarantee validity of data generated, but indicates the methodology, equipment, quality control procedures, records, and proficiency of the laboratory have been examined and found to be acceptable.

This certificate shall be valid until December 31, 2006

Certificate No. 627

James W. Meyer

OKLAHOMA
DEPARTMENT OF ENVIRONMENTAL QUALITY

State Environmental Laboratory
P.O. Box 1677
Oklahoma City, Oklahoma 73101-1677
405-702-1000

Analytical Management Laboratories,
ID # 9966
Tenkasi S. Viswanathan
15130 South Keeler
Olathe, KS 66062
(913) 829-0101

Laboratory Accreditation Program
General Water Quality\Sludge Testing
Certified parameters from 9-1-2005 to 8-31-2006

Metals

ALUMINUM	ANTIMONY	ARSENIC
BARIUM	BERYLLIUM	CADMIUM
CALCIUM	CHROMIUM	COBALT
COPPER	IRON	LEAD
MAGNESIUM	MANGANESE	MERCURY
MOLYBDENUM	NICKEL	SELENIUM
SILVER	SODIUM	THALLIUM
TIN	VANADIUM	ZINC

Nutrients

AMMONIA-NITROGEN	NITRATE-NITROGEN	NITRATE-NITRITE-NITROGEN
NITRITE-NITROGEN	ORTHOPHOSPHATE PHOSPHORUS	TOTAL PHOSPHORUS

OKLAHOMA
DEPARTMENT OF ENVIRONMENTAL QUALITY
...for a clear, attractive, prosperous Oklahoma

Demands

5-DAY BOD	CARBONACEOUS BOD	COD
TOC		

Extractable Organics

2-NITROPHENOL	4-NITROPHENOL	N-NITROSODIMETHYLAMINE
N-NITROSODI-N-PROPYLAMINE	ACENAPHTHENE	ACENAPHTHYLENE
ANTHRACENE	BENZIDINE	BENZOIC ACID
BENZO(A)ANTHRACENE	BENZO(A)PYRENE	BENZO(B)FLUORANTHENE
BENZO(G,H,I)PERYLENE	BENZO(K)FLUORANTHENE	BENZYL BUTYL PHTHALATE
BIS(2-CHLOROETHOXY)METHANE	BIS(2-CHLOROETHYL)ETHER	BIS(2-ETHYLHEXYL)PHTHALATE
4-BROMOPHENYLPHENYLETHER	CARBAZOLE	4-CHLORO-3-METHYLPHENOL
2-CHLORONAPHTHALENE	2-CHLOROPHENOL	4-CHLOROPHENYL PHENYL ETHER
CHRYSENE	DIBENZO(A,H)ANTHRACENE	DIBENZOFURAN

OKLAHOMA
DEPARTMENT OF ENVIRONMENTAL QUALITY

State Environmental Laboratory
P.O. Box 1677
Oklahoma City, Oklahoma 73101-1677
405-702-1000

Analytical Management Laboratories,
ID # 9966
Tenkasi S. Viswanathan
15130 South Keeler
Olathe, KS 66062
(913) 829-0101

Laboratory Accreditation Program
General Water Quality\Sludge Testing
Certified parameters from 9-1-2005 to 8-31-2006

Extractable Organics

1,2-DICHLOROBENZENE	1,3-DICHLOROBENZENE	1,4-DICHLOROBENZENE
3,3'-DICHLOROBENZIDINE	DIETHYL PHTHALATE	2,4-DICHLOROPHENOL
2,4-DIMETHYLPHENOL	DIMETHYL PHTHALATE	DI-N-BUTYL PHTHALATE
DI-N-OCTYL PHTHALATE	2,4-DINITROPHENOL	2,4-DINITROTOLUENE
2,6-DINITROTOLUENE	DIPHENYLAMINE	DIPHENYL ETHER
1,2-DIPHENYLHYDRAZINE	FLUORANTHENE	FLUORENE
HEXACHLOROBENZENE	HEXACHLOROBUTADIENE	HEXACHLOROCYCLOPENTADIENE
HEXACHLOROETHANE	INDENO(1,2,3-CD)PYRENE	ISOPHORONE
2-METHYL-4,6-DINITROPHENOL	NAPHTHALENE	B-NAPHTHYLAMINE
NITROBENZENE	N-NITROSODIPHENYLAMINE	PENTACHLOROPHENOL
PHENANTHRENE	PHENOL	PYRENE
1,2,3-TRICHLOROBENZENE	1,2,4-TRICHLOROBENZENE	2,3,5-TRICHLOROPHENOL
2,4,5-TRICHLOROPHENOL	2,4,6-TRICHLOROPHENOL	

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General Chemistry I

ACIDITY	ALKALINITY	BROMIDE
CALCIUM	CHLORIDE	COPPER
FLUORIDE	HARDNESS	HYDROGEN ION (PH)
IRON	MAGNESIUM	MANGANESE
NITRATE	TOTAL RESIDUE	TOTAL DISSOLVED SOLIDS
TOTAL SUSPENDED SOLIDS	SETTLEABLE RESIDUE	VOLATILE RESIDUE
SULFATE	TURBIDITY	ZINC

General Chemistry II

HEXAVALENT-CHROMIUM	TOTAL RESIDUAL CHLORINE	TOTAL CYANIDE
CYANIDE AMENABLE TO CHLORINATION	OIL AND GREASE	DISSOLVED OXYGEN

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General Chemistry II

PHENOLICS	SPECIFIC CONDUCTANCE	SULFIDE

Pesticides-Herbicides-PCB's

HEPTACHLOR	HEPTACHLOR EPOXIDE	METHOXYCHLOR
TOXAPHENE	2,4-D	2,4-DB
DALAPON	DICAMBA	DICHLORPROP
DINOSEB	2,4,5-T	2,4,5-TP
MCPA	MCPP	PCB-1016
PCB-1221	ALDRIN	ALPHA-BHC
BETA-BHC	DELTA-BHC	GAMMA-BHC (LINDANE)
CHLORDANE	4,4'-DDD	4,4'-DDE
4,4'-DDT	DIBROMOCHLOROPROPANE	DIELDRIN
ENDOSULFAN I	ENDOSULFAN II	ENDOSULFAN SULFATE
ENDRIN	ENDRIN ALDEHYDE	PCB-1232
PCB-1242	PCB-1248	PCB-1254
PCB-1260	ETHYLENEDIBROMIDE (EDB)	

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Purgeable Organics

ACETONE	ACROLEIN	ACRYLONITRILE
BENZENE	BROMODICHLOROMETHANE	BROMOMETHANE
BROMOFORM	CARBON TETRACHLORIDE	CHLOROETHANE
CHLOROBENZENE	2-CHLOROETHYLVINYL ETHER	CHLOROFORM
CHLOROMETHANE	DIBROMOCHLOROBENZENE	DIBROMOCHLOROMETHANE
1,2-DIBROMOETHANE (EDB)	DICHLOROBENZENE	1,2-DICHLOROBENZENE
1,3-DICHLOROBENZENE	1,4-DICHLOROBENZENE	DICHLORODIFLUOROMETHANE
1,1-DICHLOROETHANE	1,2-DICHLOROETHANE	1,1-DICHLOROETHENE
TRANS-1,2-DICHLOROETHENE	1,2-DICHLOROPROPANE	CIS-1,3-DICHLOROPROPENE

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Purgeable Organics

TRANS-1,3-DICHLOROPROPENE	DIETHYL ETHER	P-DIOXANE
ETHYLBENZENE	METHYLENE CHLORIDE	METHYL ETHYL KETONE
1,1,2,2-TETRACHLOROETHANE	TETRACHLOROETHENE	TOLUENE
1,1,1-TRICHLOROETHANE	1,1,2-TRICHLOROETHANE	TRICHLOROETHENE
TRICHLOROFLUOROMETHANE	VINYL CHLORIDE	METHYL TERTIARY BUTYL ETHER (MTBE)

Hazardous Waste Characterization

WASTE IGNITABILITY	WASTE CORROSIVITY	WASTE REACTIVITY
TCLP		

Petroleum Hydrocarbons

Soil-TOTAL PETROLEUM HYDROCARBON -OK 8020/8015 GRO	Soil-BTEX -OK 8020/8015 MOD	FLASH POINT -EPA 1010
FLASH POINT -EPA 1020A	METHYL TERT-BUTYL ETHER -EPA 8260B	Water-BTEX -OK8020/8015 MOD
Water -TOTAL PETROLEUM HYDROCARBON-OK8020/8015 GRO		

O K L A H O M A
DEPARTMENT OF ENVIRONMENTAL QUALITY
. . . for a clean, attractive, prosperous Oklahoma

Each certified laboratory shall provide a copy of this list upon request.

D H E C

PROMOTE PROTECT PROSPER

South Carolina Department of Health and Environmental Control

Environmental Laboratory Certification Program

In accordance with the provisions of Regulation 61 - 81, entitled "State Environmental Laboratory Certification Regulation"

ANALYTICAL MANAGEMENT LABS INC
15130 S KEELER RD
OLATHE, KANSAS 66062

is hereby certified to perform analyses as documented on the attached parameter list(s). This certification does not guarantee validity of data generated, but indicates the laboratory's adherence to prescribed methodology, quality control, records keeping, and reporting procedures. This certificate is the property of S.C. DHEC and must be surrendered upon demand. This certificate is non-transferable and is valid only for the parameters and methodology listed on the attached parameter list(s).

Laboratory Director: BILL L SAID
Certifying Authority: KS
Date of Issue: September 21, 2005
Date of Expiration: April 30, 2006
Certificate Number: 76003001

Carol F. Smith

Director
Office of Environmental Laboratory Certification

STATE OF KANSAS

DEPARTMENT OF HEALTH AND ENVIRONMENT

CERTIFICATE

This is to certify that Certificate No. E-10254

Analytical Management Laboratories Inc
15130 South Keeler Suite B
Olathe, KS 66062-2760

has been accredited in accordance with K.S.A. 65-1,109a for performing environmental analyses for the parameters listed on the attached form. Continuous accreditation depends on successful, ongoing participation in the program. Clients are urged to verify with this agency the laboratory's certification status for particular methods and analytes.

EFFECTIVE DATE: 05/01/2005 EXPIRATION DATE: 04/30/2006

Secretary
Department of Health and Environment

Environmental Laboratory Certification Officers

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LITHO. IN U.S.A.

KANSAS DEPARTMENT OF HEALTH AND ENVIRONMENT
ENVIRONMENTAL LABORATORY CERTIFICATION
SOLID/HAZARDOUS WASTE CERTIFICATION - PARAMETER LIST

This certificate supersedes all previous certificates

Analytical Management Laboratories Inc
15130 South Keeler Suite B
Olathe, KS 66062-2760

Certificate Number: E-10254
Effective Date: 01/03/2006
Expiration Date: 04/30/2006
Reciprocity:

The laboratory listed above is hereby approved for environmental laboratory certification in accordance with K.S.A. 65-1,109a for performing solids and/or hazardous waste analysis for the following parameters:

****CHARACTERISTICS**
Corrosivity {EPA 1110}
Extraction Procedure Toxicity {EPA 1310A}
Ignitability {EPA 1010}
Ignitability {EPA 1020A}
Mobile Metal Concentration in Oily Waste {EPA 1330A}
Multiple Extraction Procedure {EPA 1320}
Reactive Cyanide {CHAP 7, SEC. 7.3}
Reactive Sulfide {CHAP 7, SEC. 7.3}
Synthetic Precipitation Leaching Procedure {EPA 1312}
Toxic Characteristic Leaching Procedure {EPA 1311}

****DEMANDS**
Total Organic Carbon {EPA 9060}

****INORGANIC**
Cyanide {EPA 9014}
Cyanide, Amenable to CL {EPA 9010B}
Cyanide, Amenable to CL {EPA 9012A}
Cyanide, Total {EPA 9010B}
Cyanide, Total {EPA 9012A}
Extractable Cyanide {EPA 9013}

****METALS**
Aluminum {EPA 6010B}
Aluminum {EPA 6020}
Antimony {EPA 6010B}
Antimony {EPA 6020}
Arsenic {EPA 6010B}
Arsenic {EPA 6020}
Barium {EPA 6010B}
Barium {EPA 6020}
Beryllium {EPA 6010B}
Beryllium {EPA 6020}
Cadmium {EPA 6010B}
Cadmium {EPA 6020}
Calcium {EPA 6010B}
Chromium {EPA 6010B}
Chromium {EPA 6020}
Chromium, VI {EPA 7195}
Chromium, VI {EPA 7196A}
Cobalt {EPA 6010B}
Cobalt {EPA 6020}
Copper {EPA 6010B}
Copper {EPA 6020}
Iron {EPA 6010B}
Lead {EPA 6010B}
Lead {EPA 6020}
Magnesium {EPA 6010B}

NELAP-Recognized

Manganese {EPA 6010B}
Manganese {EPA 6020}
Mercury {EPA 7471A}
Molybdenum {EPA 6010B}
Nickel {EPA 6010B}
Nickel {EPA 6020}
Potassium {EPA 6010B}
Selenium {EPA 6010B}
Silver {EPA 6010B}
Silver {EPA 6020}
Sodium {EPA 6010B}
Thallium {EPA 6010B}
Thallium {EPA 6020}
Vanadium {EPA 6010B}
Zinc {EPA 6010B}
Zinc {EPA 6020}

**MINERALS
Bromide {EPA 9056}
Chloride {EPA 9056}
Fluoride {EPA 9056}
Purgeable Sulfides {EPA 9031}
Sulfate {EPA 9035}
Sulfate {EPA 9056}
Sulfide {EPA 9030B}
Sulfide {EPA 9034}

**MISCELLANEOUS
Cation-Exchange Capacity of Soils {EPA 9080}
Cation-Exchange Capacity of Soils {EPA 9081}
Hydrogen Ion (pH) {EPA 9040B}
Hydrogen Ion (pH) {EPA 9041A}
Hydrogen Ion (pH) {EPA 9045C}
Liquid Release Test Procedure (LRT) {EPA 9096}
Oil & Grease {EPA 9070A}
Oil & Grease {EPA 9071B}
Paint Filter Liquids Test {EPA 9095A}
Phenolics {EPA 9065}
Specific Conductance {EPA 9050A}
Total Chlorine in New & Used Petroleum Products {EPA 9075}

**NUTRIENTS
Nitrate {EPA 9056}
Nitrite {EPA 9056}
Phosphate, Ortho {EPA 9056}

NELAP-Recognized

**ORGANIC CHEMISTRY VOLATILES (MEASUREMENT BY GC)
{EPA 8015B} Ethylene glycol

**ORGANIC CHEMISTRY VOLATILES (MEASUREMENT BY GC/MS)
{EPA 8260B} 1,1-Dichloroethane
{EPA 8260B} 1,1-Dichloroethylene
{EPA 8260B} 1,1-Dichloropropene
{EPA 8260B} 1,1,1-Trichloroethane
{EPA 8260B} 1,1,1,2-Tetrachloroethane
{EPA 8260B} 1,1,2-Trichloroethane
{EPA 8260B} 1,1,2,2-Tetrachloroethane
{EPA 8260B} 1,2-Dibromoethane (EDB, Ethylene dibromide)
{EPA 8260B} 1,2-Dichlorobenzene
{EPA 8260B} 1,2-Dichloroethane
{EPA 8260B} 1,2-Dichloropropane

{EPA 8260B} 1,2,3-Trichlorobenzene
{EPA 8260B} 1,2,3-Trichloropropane
{EPA 8260B} 1,2,4-Trichlorobenzene
{EPA 8260B} 1,2,4-Trimethylbenzene
{EPA 8260B} 1,3-Dichlorobenzene
{EPA 8260B} 1,3-Dichloropropane
{EPA 8260B} 1,3,5-Trimethylbenzene
{EPA 8260B} 1,4-Dichlorobenzene
{EPA 8260B} 1,4-Dioxane
{EPA 8260B} 2-Chloroethyl vinyl ether
{EPA 8260B} 2-Chlorotoluene
{EPA 8260B} 2-Hexanone
{EPA 8260B} 2-Pentanone
{EPA 8260B} 2,2-Dichloropropane
{EPA 8260B} 4-Chlorotoluene
{EPA 8260B} 4-Isopropyltoluene
{EPA 8260B} 4-Methyl-2-Pentanone (MIBK)
{EPA 8260B} Acetone
{EPA 8260B} Acetonitrile
{EPA 8260B} Acrolein
{EPA 8260B} Acrylonitrile
{EPA 8260B} Benzene
{EPA 8260B} Bromobenzene
{EPA 8260B} Bromochloromethane
{EPA 8260B} Bromodichloromethane
{EPA 8260B} Bromoform
{EPA 8260B} Bromomethane
{EPA 8260B} Carbon disulfide
{EPA 8260B} Carbon Tetrachloride
{EPA 8260B} Chlorobenzene
{EPA 8260B} Chloroethane
{EPA 8260B} Chloroform
{EPA 8260B} Chloromethane
{EPA 8260B} cis-1,2-Dichloroethylene
{EPA 8260B} cis-1,3-Dichloropropene
{EPA 8260B} Dibromochloromethane
{EPA 8260B} Dibromofluoromethane
{EPA 8260B} Dibromomethane
{EPA 8260B} Dichlorodifluoromethane
{EPA 8260B} Dichloromethane (Methylene chloride)
{EPA 8260B} Diethyl ether
{EPA 8260B} Ethyl methacrylate
{EPA 8260B} Ethylbenzene
{EPA 8260B} Hexachlorobutadiene
{EPA 8260B} Hexachloroethane
{EPA 8260B} Iodomethane
{EPA 8260B} Isobutyl Alcohol
{EPA 8260B} Isopropylbenzene
{EPA 8260B} meta-Xylene
{EPA 8260B} Methacrylonitrile
{EPA 8260B} Methyl acrylate
{EPA 8260B} Methyl ethyl ketone
{EPA 8260B} Methyl tert-butyl ether (MTBE)
{EPA 8260B} Methylmethacrylate
{EPA 8260B} n-Butyl alcohol
{EPA 8260B} n-Butylbenzene
{EPA 8260B} n-Propylbenzene
{EPA 8260B} Naphthalene
{EPA 8260B} ortho-Xylene
{EPA 8260B} para-Xylene
{EPA 8260B} Propionitrile

NELAP-Recognized

{EPA 8260B} Pyridine
{EPA 8260B} sec-Butylbenzene
{EPA 8260B} Styrene
{EPA 8260B} t-Butyl alcohol
{EPA 8260B} tert-Butylbenzene
{EPA 8260B} Tetrachloroethylene
{EPA 8260B} Toluene
{EPA 8260B} trans-1,2-Dichloroethylene
{EPA 8260B} trans-1,3-Dichloropropene
{EPA 8260B} trans-1,4-Dichloro-2-butene
{EPA 8260B} Trichloroethylene
{EPA 8260B} Trichlorofluoromethane
{EPA 8260B} Vinyl Acetate
{EPA 8260B} Vinyl Chloride

**ORGANIC CHEMISTRY (MEASUREMENT BY GC)
{EPA 8081A} 1,2-Dibromo-3-chloropropane (DBCP)
{EPA 8081A} 4,4'-DDD
{EPA 8081A} 4,4'-DDE
{EPA 8081A} 4,4'-DDT
{EPA 8081A} Alachlor
{EPA 8081A} Aldrin
{EPA 8081A} alpha-BHC (alpha-Hexachlorocyclohexane)
{EPA 8081A} alpha-Chlordane
{EPA 8081A} beta-BHC (beta-Hexachlorocyclohexane)
{EPA 8081A} Chlordane (Tech)
{EPA 8081A} delta-BHC
{EPA 8081A} Dieldrin
{EPA 8081A} Endosulfan I
{EPA 8081A} Endosulfan II
{EPA 8081A} Endosulfan Sulfate
{EPA 8081A} Endrin
{EPA 8081A} Endrin aldehyde
{EPA 8081A} Endrin ketone
{EPA 8081A} g-Chlordane
{EPA 8081A} gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)
{EPA 8081A} Heptachlor
{EPA 8081A} Heptachlor Epoxide
{EPA 8081A} Methoxychlor
{EPA 8081A} Mirex
{EPA 8081A} Propachlor (Ramrod)
{EPA 8081A} Toxaphene (Chlorinated camphene)
{EPA 8082} PCB-1016
{EPA 8082} PCB-1221
{EPA 8082} PCB-1232
{EPA 8082} PCB-1242
{EPA 8082} PCB-1248
{EPA 8082} PCB-1254
{EPA 8082} PCB-1260
{EPA 8151A} 2,4-D
{EPA 8151A} 2,4-DB
{EPA 8151A} 2,4,5-T
{EPA 8151A} 2,4,5-TP
{EPA 8151A} 3,5-Dichlorobenzoic acid
{EPA 8151A} Aciflurofen
{EPA 8151A} Bentazon
{EPA 8151A} Dalapon
{EPA 8151A} DCPA Di-Acid
{EPA 8151A} Dicamba
{EPA 8151A} Dichlorprop
{EPA 8151A} Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)

NELAP-Recognized

{EPA 8151A} MCPA
{EPA 8151A} Pentachlorophenol
{EPA 8151A} Picloram

**ORGANIC CHEMISTRY (MEASUREMENT BY GC/MS)
{EPA 8270C} 1,2-Dichlorobenzene
{EPA 8270C} 1,2-Dinitrobenzene
{EPA 8270C} 1,2-Diphenylhydrazine
{EPA 8270C} 1,2,4-Trichlorobenzene
{EPA 8270C} 1,2,4,5-Tetrachlorobenzene
{EPA 8270C} 1,3-Dinitrobenzene
{EPA 8270C} 1,3,5-Trinitrobenzene
{EPA 8270C} 1,4-Dichlorobenzene
{EPA 8270C} 2-Chloronaphthalene
{EPA 8270C} 2-Chlorophenol
{EPA 8270C} 2-Methyl-4,6-Dinitrophenol
{EPA 8270C} 2-Methylnaphthalene
{EPA 8270C} 2-Methylphenol (o-Cresol)
{EPA 8270C} 2-Naphthylamine
{EPA 8270C} 2-Nitroaniline
{EPA 8270C} 2-Nitrophenol
{EPA 8270C} 2,3,4,6-Tetrachlorophenol
{EPA 8270C} 2,4-Dichlorophenol
{EPA 8270C} 2,4-Dimethylphenol
{EPA 8270C} 2,4-Dinitrophenol
{EPA 8270C} 2,4-Dinitrotoluene (2,4-DNT)
{EPA 8270C} 2,4,5-Trichlorophenol
{EPA 8270C} 2,4,6-Trichlorophenol
{EPA 8270C} 2,6-Dichlorophenol
{EPA 8270C} 2,6-Dinitrotoluene
{EPA 8270C} 3-Methylphenol (m-Cresol)
{EPA 8270C} 3-Nitroaniline
{EPA 8270C} 3,3'-Dichlorobenzidine
{EPA 8270C} 3,3'-Dimethoxybenzidine
{EPA 8270C} 4-Bromophenyl phenyl ether
{EPA 8270C} 4-Chloro-3-methylphenol
{EPA 8270C} 4-Chloroaniline
{EPA 8270C} 4-Chlorophenyl phenyl ether
{EPA 8270C} 4-Methylphenol (p-Cresol)
{EPA 8270C} 4-Nitroaniline
{EPA 8270C} 4-Nitrophenol
{EPA 8270C} Acenaphthene
{EPA 8270C} Acenaphthylene
{EPA 8270C} Acetophenone
{EPA 8270C} Aniline
{EPA 8270C} Anthracene
{EPA 8270C} Benzidine
{EPA 8270C} Benzoic acid
{EPA 8270C} Benzo(a)anthracene
{EPA 8270C} Benzo(a)pyrene
{EPA 8270C} Benzo(b)fluoranthene
{EPA 8270C} Benzo(g,h,i)perylene
{EPA 8270C} Benzo(k)fluoranthene
{EPA 8270C} Benzyl alcohol
{EPA 8270C} Bis(2-chloroethoxy)methane
{EPA 8270C} Bis(2-chloroethyl)ether
{EPA 8270C} Bis(2-chloroisopropyl)ether
{EPA 8270C} Bis(2-Ethylhexyl) Phthalate
{EPA 8270C} Butyl benzyl phthalate
{EPA 8270C} Chlorobenzilate
{EPA 8270C} Chrysene

CREDITING AUTHORITY nelap
NELAP-Recognized

{EPA 8270C} Di-n-butyl phthalate
{EPA 8270C} Di-n-octyl phthalate
{EPA 8270C} Dibenzofuran
{EPA 8270C} Dibenzo(a,h)anthracene
{EPA 8270C} Diethyl phthalate
{EPA 8270C} Dimethyl phthalate
{EPA 8270C} Diphenylamine
{EPA 8270C} Ethyl methanesulfonate
{EPA 8270C} Fluoranthene
{EPA 8270C} Fluorene
{EPA 8270C} Hexachlorobenzene
{EPA 8270C} Hexachlorobutadiene
{EPA 8270C} Hexachlorocyclopentadiene
{EPA 8270C} Hexachloroethane
{EPA 8270C} Indeno(1,2,3-c,d)pyrene
{EPA 8270C} Isophorone
{EPA 8270C} Methylmethanesulfonate
{EPA 8270C} N-Nitrosodi-n-butylamine
{EPA 8270C} N-Nitrosodi-n-propylamine
{EPA 8270C} N-Nitrosodiethylamine
{EPA 8270C} N-Nitrosodimethylamine
{EPA 8270C} N-Nitrosodiphenylamine
{EPA 8270C} Naphthalene
{EPA 8270C} Nitrobenzene
{EPA 8270C} Pentachlorobenzene
{EPA 8270C} Pentachlorophenol
{EPA 8270C} Phenacetin
{EPA 8270C} Phenanthrene
{EPA 8270C} Phenol
{EPA 8270C} Pyrene
{EPA 8270C} Pyridine

**ORGANIC CHEMISTRY (MEASUREMENT BY HPLC)
{EPA 8310} Acenaphthene
{EPA 8310} Acenaphthylene
{EPA 8310} Anthracene
{EPA 8310} Benzo(a)anthracene
{EPA 8310} Benzo(a)pyrene
{EPA 8310} Benzo(b)fluoranthene
{EPA 8310} Benzo(g,h,i)perylene
{EPA 8310} Benzo(k)fluoranthene
{EPA 8310} Chrysene
{EPA 8310} Dibenzo(a,h)anthracene
{EPA 8310} Fluoranthene
{EPA 8310} Fluorene
{EPA 8310} Indeno(1,2,3-c,d)pyrene
{EPA 8310} Naphthalene
{EPA 8310} Phenanthrene
{EPA 8310} Pyrene
{EPA 8330} 1,3-Dinitrobenzene
{EPA 8330} 1,3,5-Trinitrobenzene
{EPA 8330} 2-Amino-4,6-dinitrotoluene
{EPA 8330} 2,4-Dinitrotoluene (2,4-DNT)
{EPA 8330} 2,4,6-Trinitrotoluene
{EPA 8330} 2,6-Dinitrotoluene
{EPA 8330} 4-Amino-2,6-dinitrotoluene
{EPA 8330} HMX
{EPA 8330} meta-Nitrotoluene
{EPA 8330} Methyl-2,4,6-trinitrophenylnitramine
{EPA 8330} Nitrobenzene
{EPA 8330} ortho-Nitrotoluene

ACCREDITING nelap AUTHORITY
NELAP-Recognized

{EPA 8330} para-Nitrotoluene
{EPA 8330} RDX
{EPA 8332} Nitroglycerine

****Total Petroleum Hydrocarbons**
{EPA 8015B} DRO
{EPA 8015B} GRO

End of Parameter List

NELAP-Recognized

KANSAS DEPARTMENT OF HEALTH AND ENVIRONMENT
ENVIRONMENTAL LABORATORY CERTIFICATION
WASTE WATER CERTIFICATION - PARAMETER LIST

This certificate supersedes all previous certificates

Analytical Management Laboratories Inc
15130 South Keeler Suite B
Olathe, KS 66062-2760

Certificate Number: E-10254
Effective Date: 07/08/2005
Expiration Date: 04/30/2006
Reciprocity:

The laboratory listed above is hereby approved for environmental laboratory certification in accordance with K.S.A. 65-1,109a for performing waste water analysis for the following parameters:

****DEMANDS**
BOD {EPA 405.1}
BOD {SM 5210B}
cBOD {SM 5210B}
Oxygen, Dissolved {EPA 360.1}

****INORGANIC**
Chlorine - total {EPA 330.4}
Cyanide, Amenable to CL {EPA 335.1}
Cyanide, Amenable to CL {SM 4500-CN C}
Cyanide, Amenable to CL {SM 4500-CN G}
Cyanide, Total {EPA 335.2}
Cyanide, Total {SM 4500-CN C}
Cyanide, Total {SM 4500-CN E}

****METALS**
Aluminum {EPA 200.7}
Aluminum {EPA 200.8 - ATP}
Antimony {EPA 200.7}
Antimony {EPA 200.8 - ATP}
Arsenic {EPA 200.7}
Arsenic {EPA 200.8 - ATP}
Barium {EPA 200.7}
Barium {EPA 200.8 - ATP}
Beryllium {EPA 200.7}
Beryllium {EPA 200.8 - ATP}
Cadmium {EPA 200.7}
Cadmium {EPA 200.8 - ATP}
Calcium {EPA 200.7}
Chromium {EPA 200.7}
Chromium {EPA 200.8 - ATP}
Cobalt {EPA 200.7}
Copper {EPA 200.7}
Iron {EPA 200.7}
Lead {EPA 200.7}
Lead {EPA 200.8 - ATP}
Magnesium {EPA 200.7}
Manganese {EPA 200.7}
Mercury {EPA 245.1}
Mercury {EPA 245.2}
Molybdenum {EPA 200.7}
Nickel {EPA 200.7}
Nickel {EPA 200.8 - ATP}
Potassium {EPA 200.7}
Selenium {EPA 200.7}
Selenium {EPA 200.8 - ATP}
Silver {EPA 200.7}
Silver {EPA 200.8 - ATP}
Sodium {EPA 200.7}
Thallium {EPA 200.7}

NELAP-Recognized

Thallium {EPA 200.8 - ATP}
Tin {EPA 200.8 - ATP}
Vanadium {EPA 200.7}
Vanadium {EPA 200.8 - ATP}
Zinc {EPA 200.7}
Zinc {EPA 200.8 - ATP}

**METALS - 503 Regs
Arsenic {EPA 6010B}
Arsenic {EPA 6020}
Cadmium {EPA 6010B}
Cadmium {EPA 6020}
Chromium, Total {EPA 6010B}
Chromium, Total {EPA 6020}
Copper {EPA 6010B}
Copper {EPA 6020}
Lead {EPA 6010B}
Lead {EPA 6020}
Mercury {EPA 6020}
Mercury {EPA 7470}
Mercury {EPA 7471}
Molybdenum {EPA 6010B}
Molybdenum {EPA 6020}
Nickel {EPA 6010B}
Nickel {EPA 6020}
Selenium {EPA 6010B}
Selenium {EPA 6020}
Zinc {EPA 6010B}
Zinc {EPA 6020}

**MINERALS
Acidity {EPA 305.1}
Acidity {SM 2310B}
Alkalinity {EPA 310.1}
Alkalinity {SM 2320B}
Chloride {EPA 300.0}
Chloride {EPA 325.3}
Fluoride {EPA 300.0}
Hardness {SM 2340B}
Sulfate {EPA 300.0}
Sulfate {EPA 375.4}
Sulfide {EPA 376.2}
Sulfide {SM 4500-S-2 D}
Sulfite {EPA 377.1}
Sulfite {SM 4500-SO3-2 B}

NELAP-Recognized

**MISCELLANEOUS
Bromide {EPA 300.0}
Color {EPA 110.2}
Hydrogen Ion (pH) {EPA 150.1}
Oil & Grease {EPA 1664}
Oil & Grease {SM 5520B}
Phenolics {EPA 420.1}
Surfactants (MBAS) {EPA 425.1}
Temperature {EPA 170.1}
Turbidity {EPA 180.1}

**NUTRIENTS
Ammonia {EPA 350.3}
Nitrate-Nitrite {EPA 300.0}
Nitrate-Nitrite {EPA 353.3}

Nitrate {EPA 300.0}
Nitrate {EPA 353.3}
Nitrite {EPA 300.0}
Nitrite {EPA 354.1}
O-Phosphate {EPA 300.0}
O-Phosphate {EPA 365.3}
Phosphorus {EPA 365.3}
Phosphorus {SM 4500-P E}

**ORGANIC CHEMISTRY VOLATILES (MEASUREMENT BY GC/MS)
{EPA 624} 1,1-Dichloroethane
{EPA 624} 1,1-Dichloroethylene
{EPA 624} 1,1,1-Trichloroethane
{EPA 624} 1,1,2-Trichloroethane
{EPA 624} 1,1,2,2-Tetrachloroethane
{EPA 624} 1,2-Dichloroethane
{EPA 624} 1,2-Dichloropropane
{EPA 624} 2-Chloroethyl vinyl ether
{EPA 624} Acrolein
{EPA 624} Acrylonitrile
{EPA 624} Benzene
{EPA 624} Bromodichloromethane
{EPA 624} Bromoform
{EPA 624} Bromomethane
{EPA 624} Carbon Tetrachloride
{EPA 624} Chlorobenzene
{EPA 624} Chloroethane
{EPA 624} Chloroform
{EPA 624} Chloromethane
{EPA 624} cis-1,3-Dichloropropene
{EPA 624} Dibromochloromethane
{EPA 624} Dichloromethane (Methylene chloride)
{EPA 624} Ethylbenzene
{EPA 624} Tetrachloroethylene
{EPA 624} Toluene
{EPA 624} trans-1,2-Dichloroethylene
{EPA 624} trans-1,3-Dichloropropene
{EPA 624} Trichloroethylene
{EPA 624} Trichlorofluoromethane
{EPA 624} Vinyl Chloride

**ORGANIC CHEMISTRY (MEASUREMENT BY GC)
{EPA 608} 4,4'-DDD
{EPA 608} 4,4'-DDE
{EPA 608} 4,4'-DDT
{EPA 608} Aldrin
{EPA 608} alpha-BHC (alpha-Hexachlorocyclohexane)
{EPA 608} beta-BHC (beta-Hexachlorocyclohexane)
{EPA 608} Chlordane
{EPA 608} delta-BHC
{EPA 608} Dieldrin
{EPA 608} Endosulfan I
{EPA 608} Endosulfan II
{EPA 608} Endosulfan Sulfate
{EPA 608} Endrin
{EPA 608} Endrin aldehyde
{EPA 608} gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)
{EPA 608} Heptachlor
{EPA 608} Heptachlor epoxide
{EPA 608} PCB-1016
{EPA 608} PCB-1232

ACCREDITING AUTHORITY nelap
NELAP-Recognized

{EPA 608} PCB-1242
{EPA 608} PCB-1248
{EPA 608} PCB-1254
{EPA 608} PCB-1260
{EPA 608} Toxaphene (Chlorinated camphene)
{SM 6630C} 4,4'-DDD
{SM 6630C} 4,4'-DDE
{SM 6630C} 4,4'-DDT
{SM 6630C} Aldrin
{SM 6630C} alpha-BHC (alpha-Hexachlorocyclohexane)
{SM 6630C} Aroclor-1016 (PCB-1016)
{SM 6630C} Aroclor-1232 (PCB-1232)
{SM 6630C} Aroclor-1242 (PCB-1242)
{SM 6630C} Aroclor-1248 (PCB-1248)
{SM 6630C} Aroclor-1254 (PCB-1254)
{SM 6630C} Aroclor-1260 (PCB-1260)
{SM 6630C} beta-BHC (beta-Hexachlorocyclohexane)
{SM 6630C} Chlordane
{SM 6630C} delta-BHC
{SM 6630C} Dieldrin
{SM 6630C} Endosulfan I
{SM 6630C} Endosulfan II
{SM 6630C} Endosulfan Sulfate
{SM 6630C} Endrin
{SM 6630C} Endrin aldehyde
{SM 6630C} gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)
{SM 6630C} Heptachlor
{SM 6630C} Heptachlor Epoxide
{SM 6630C} Methoxychlor
{SM 6630C} Toxaphene (Chlorinated camphene)

**ORGANIC CHEMISTRY (MEASUREMENT BY GC/MS)
{EPA 625} 1,2-Dichlorobenzene
{EPA 625} 1,2,4-Trichlorobenzene
{EPA 625} 1,3-Dichlorobenzene
{EPA 625} 1,4-Dichlorobenzene
{EPA 625} 2-Chloronaphthalene
{EPA 625} 2-Chlorophenol
{EPA 625} 2-Methyl-4,6-Dinitrophenol
{EPA 625} 2-Nitrophenol
{EPA 625} 2,4-Dichlorophenol
{EPA 625} 2,4-Dimethylphenol
{EPA 625} 2,4-Dinitrophenol
{EPA 625} 2,4-Dinitrotoluene (2,4-DNT)
{EPA 625} 2,4,6-Trichlorophenol
{EPA 625} 2,6-Dinitrotoluene
{EPA 625} 3,3'-Dichlorobenzidine
{EPA 625} 4-Bromophenyl phenyl ether
{EPA 625} 4-Chloro-3-methylphenol
{EPA 625} 4-Chlorophenyl phenyl ether
{EPA 625} 4-Nitrophenol
{EPA 625} Acenaphthene
{EPA 625} Acenaphthylene
{EPA 625} Anthracene
{EPA 625} Benzidine
{EPA 625} Benzo(a)anthracene
{EPA 625} Benzo(a)pyrene
{EPA 625} Benzo(b)fluoranthene
{EPA 625} Benzo(g,h,i)perylene
{EPA 625} Benzo(k)fluoranthene
{EPA 625} Benzyl butyl phthalate

ACCREDITING AUTHORITY nelap
NELAP-Recognized

{EPA 625} Bis(2-chloroethoxy)methane
{EPA 625} Bis(2-chloroethyl)ether
{EPA 625} bis(2-Chloroisopropyl)ether
{EPA 625} Bis(2-ethylhexyl)phthalate
{EPA 625} Chrysene
{EPA 625} Di-n-butyl phthalate
{EPA 625} Di-n-octyl phthalate
{EPA 625} Dibenzo(a,h)anthracene
{EPA 625} Diethyl phthalate
{EPA 625} Dimethyl phthalate
{EPA 625} Fluoranthene
{EPA 625} Fluorene
{EPA 625} gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)
{EPA 625} Hexachlorobenzene
{EPA 625} Hexachlorobutadiene
{EPA 625} Hexachlorocyclopentadiene
{EPA 625} Hexachloroethane
{EPA 625} Indeno(1,2,3-c,d)pyrene
{EPA 625} Isophorone
{EPA 625} N-Nitrosodi-n-propylamine
{EPA 625} N-Nitrosodimethylamine
{EPA 625} N-Nitrosodiphenylamine
{EPA 625} Naphthalene
{EPA 625} Nitrobenzene
{EPA 625} Pentachlorophenol
{EPA 625} Phenanthrene
{EPA 625} Phenol
{EPA 625} Pyrene

**RESIDUES
Residue, Filterable (TDS) {EPA 160.1}
Residue, Filterable (TDS) {SM 2540C}
Residue, Non Filterable (TSS) {EPA 160.2}
Residue, Non Filterable (TSS) {SM 2540D}
Residue, Settleable {EPA 160.5}
Residue, Settleable {SM 2540F}
Residue, Total {EPA 160.3}
Residue, Total {SM 2540B}
Residue, Volatile {EPA 160.4}

End of Parameter List

NELAP-Recognized

KANSAS DEPARTMENT OF HEALTH AND ENVIRONMENT
ENVIRONMENTAL LABORATORY CERTIFICATION
DRINKING WATER CERTIFICATION - PARAMETER LIST

This certificate supersedes all previous certificates

Analytical Management Laboratories Inc
15130 South Keeler Suite B
Olathe, KS 66062-2760

Certificate Number: E-10254
Effective Date: 05/01/2005
Expiration Date: 04/30/2006
Reciprocity:

The laboratory listed above is hereby approved for environmental laboratory certification in accordance with K.S.A. 65-1,109a for performing drinking water analysis for the following parameters:

**METALS
Aluminum {EPA 200.7}
Aluminum {EPA 200.8}
Antimony {EPA 200.8}
Arsenic {EPA 200.8}
Barium {EPA 200.7}
Barium {EPA 200.8}
Beryllium {EPA 200.7}
Beryllium {EPA 200.8}
Cadmium {EPA 200.7}
Cadmium {EPA 200.8}
Chromium {EPA 200.7}
Chromium {EPA 200.8}
Copper {EPA 200.7}
Copper {EPA 200.8}
Iron {EPA 200.7}
Lead {EPA 200.8}
Manganese {EPA 200.7}
Manganese {EPA 200.8}
Mercury {EPA 200.8}
Mercury {EPA 245.1}
Mercury {EPA 245.2}
Nickel {EPA 200.7}
Nickel {EPA 200.8}
Selenium {EPA 200.8}
Silver {EPA 200.7}
Silver {EPA 200.8}
Zinc {EPA 200.7}
Zinc {EPA 200.8}

NELAP-Recognized

**MINERALS
Chloride {EPA 300.0}
Fluoride {EPA 300.0}
Sulfate {EPA 300.0}

**NUTRIENTS
Nitrate {EPA 300.0}
Nitrate {EPA 353.2}
Nitrate {SM 4500-NO3 E}

**ORGANIC CHEMISTRY ADIPATE/PHTALATE
{EPA 525.2} Di(ethylhexyl)phthalate

**ORGANIC CHEMISTRY DBCP/EDB
{EPA 504.1} 1,2-Dibromoethane (EDB, Ethylene dibromide)
{EPA 504.1} 1,2,3-Trichloropropane

**ORGANIC CHEMISTRY HERBICIDES
{EPA 515.4} Dalapon
{EPA 515.4} Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)

{EPA 515.4} Pentachlorophenol

**ORGANIC CHEMISTRY P.A.Hs
{EPA 525.2} Benzo(a)pyrene

**ORGANIC CHEMISTRY VOLATILES
{EPA 524.2} 1,1-Dichloroethylene
{EPA 524.2} 1,1,1-Trichloroethane
{EPA 524.2} 1,1,2-Trichloroethane
{EPA 524.2} 1,2-Dichlorobenzene
{EPA 524.2} 1,2-Dichloroethane
{EPA 524.2} 1,2-Dichloropropane
{EPA 524.2} 1,4-Dichlorobenzene
{EPA 524.2} Benzene
{EPA 524.2} Carbon Tetrachloride
{EPA 524.2} Chlorobenzene
{EPA 524.2} cis-1,2-Dichloroethylene
{EPA 524.2} Dichloromethane (Methylene chloride)
{EPA 524.2} Ethylbenzene
{EPA 524.2} Styrene
{EPA 524.2} Tetrachloroethylene
{EPA 524.2} Toluene
{EPA 524.2} trans-1,2-Dichloroethylene
{EPA 524.2} Trichloroethylene
{EPA 524.2} Vinyl Chloride
{EPA 524.2} Xylene

**RESIDUES
Residue, Filterable (TDS) {SM 2540C}

**TRIHALOMETHANES
{EPA 524.2} Bromodichloromethane
{EPA 524.2} Bromoform
{EPA 524.2} Chloroform
{EPA 524.2} Dibromochloromethane

**UNREGULATED ORGANIC COMPOUNDS
{EPA 524.2} 1,1-Dichloroethane
{EPA 524.2} 1,1-Dichloropropene
{EPA 524.2} 1,1,1,2-Tetrachloroethane
{EPA 524.2} 1,1,2,2-Tetrachloroethane
{EPA 524.2} 1,2,3-Trichlorobenzene
{EPA 524.2} 1,2,4-Trimethylbenzene
{EPA 524.2} 1,3-Dichlorobenzene
{EPA 524.2} 1,3-Dichloropropane
{EPA 524.2} 1,3,5-Trimethylbenzene
{EPA 524.2} 2-Chlorotoluene
{EPA 524.2} 2,2-Dichloropropane
{EPA 524.2} 4-Chlorotoluene
{EPA 524.2} 4-Isopropyltoluene
{EPA 524.2} Bromobenzene
{EPA 524.2} Bromochloromethane
{EPA 524.2} Bromomethane
{EPA 524.2} Chloroethane
{EPA 524.2} Chloromethane
{EPA 524.2} cis-1,3-Dichloropropene
{EPA 524.2} Dibromomethane
{EPA 524.2} Dichlorodifluoromethane
{EPA 524.2} Hexachlorobutadiene
{EPA 524.2} Isopropylbenzene
{EPA 524.2} Methyl-t-butyl ether

ACCREDITING AUTHORITY nelap
NELAP-Recognized

{EPA 524.2} n-Butylbenzene
{EPA 524.2} n-Propylbenzene
{EPA 524.2} Naphthalene
{EPA 524.2} sec-Butylbenzene
{EPA 524.2} tert-Butylbenzene
{EPA 524.2} trans-1,3-Dichloropropene
{EPA 524.2} Trichlorofluoromethane

End of Parameter List

NELAP-Recognized

Performance Test Results

State of Kansas

Bill Graves Governor

Department of Health and Environment

James J. O'Connell, Secretary

MEMORANDUM

To: Participating Laboratories

From: Jack R. McKenzie and Aurora P. Shields
Laboratory Improvement Specialists

Date: September, 1995

Re: WP034 Data Evaluation

Enclosed please find an evaluation of the data which your laboratory reported for EPA Performance Evaluation (P.E.) Study WP034. Review this data carefully. Any data which has been evaluated as other than acceptable should be closely checked and the procedures used to obtain that value scrutinized carefully. The quality of the data which your laboratory is routinely producing for such a parameter may be in question.

Please respond in writing to our office concerning any corrective action your laboratory has taken on unacceptable parameters. This information may be considered when our office evaluates your parameters for future certification.

According to information received from EMSL-Cincinnati they report: "failure rate for beryllium in trace metals sample 1 (TV=13.0 µg/L) was 26.5 percent of those reporting usable data. The failure rate was somewhat higher for ICP users, probably because of the increase in variability caused by the correction for the vanadium present in the same sample. We plan to move vanadium or beryllium into trace metals sample 2 so this interference problem can be avoided, probably starting with WS039.

One of the analyte/sample combinations in the study was NOT evaluated. The TDS in minerals sample 2 was not evaluated because the variability of the study data was about three times higher than expected and there was concern that this might have resulted from TDS instability in the sample during the study. We thought it best to just ignore data reported for TDS sample 2."

If you have any questions please feel free to contact our office at (913) 296-1639 - Jack McKenzie or (913) 296-6198 - Aurora Shields.

Kansas Health and Environmental Laboratory
Forbes Field, Bldg. 740, Topeka, Kansas 66620-0001

Telephone: Jack McKenzie (913) 296-1639
Aurora Shields (913) 296-6198

Performance Evaluation Report
USEPA Water Pollution Study WP034

Date: 29AUG95

articipant ID: KS009C2 Type: OTHER Requesting Office: RS

Sample Number	Reported Value	True Value*	Acceptance Limits	Warning Limits	Performance Evaluation
TRACE METALS IN MICROGRAMS/LITER					
01-ALUMINUM					
C1	930.	990	830- 1140	868- 1100	Accept.
C2	2430	2700	2290- 3060	2390- 2960	Accept.
02-ARSENIC					
C1	141.	121	98.4- 144	104- 138	Ck. for Err.
C2	499.	462	380- 544	400- 523	Accept.
03-BERYLLIUM					
C1	13.5	13.0	9.16- 16.8	10.1- 15.8	Accept.
C2	92.0	92.3	77.1- 105	80.6- 102	Accept.
04-CADMIUM					
C1	12.1	13.0	10.2- 16.1	10.9- 15.4	Accept.
C2	197.	210	181- 240	188- 232	Accept.
05-COBALT					
C1	133.	130	114- 145	118- 141	Accept.
C2	330.	330	293- 369	303- 359	Accept.
06-CHROMIUM					
C1	99.1	97.2	82.9- 111	86.5- 108	Accept.
C2	357.	361	312- 409	324- 396	Accept.
07-COPPER					
C1	53.0	50.4	41.6- 58	43.6- 56	Accept.
C2	930.	890	792- 980	816- 957	Accept.
08-IRON					
C1	595.	626	555- 700	573- 682	Accept.
C2	1010	941	834- 1070	863- 1040	Accept.
09-MERCURY					
C1	1.31	1.33	0.929- 1.77	1.03- 1.66	Accept.
C2	2.00	1.76	1.26- 2.3	1.39- 2.17	Accept.
10-MANGANESE					
C1	290.	281	251- 310	258- 303	Accept.
C2	1465	1400	1260- 1540	1290- 1500	Accept.
11-NICKEL					
C1	86.0	80.9	67.7- 91.9	70.7- 88.9	Accept.
C2	820.	780	701- 860	721- 840	Accept.
12-LEAD					
C1	194.	190	162- 218	169- 211	Accept.
C2	521.	500	438- 561	453- 545	Accept.
13-SELENIUM					
C1	183.	199	146- 239	158- 227	Accept.
C2	422.	437	322- 524	347- 498	Accept.
14-VANADIUM					
C1	5405.	5200	4600- 5730	4750- 5590	Accept.
C2	9590.	9100	8060-10000	8310- 9770	Accept.
15-ZINC					
C1	510.	484	428- 541	443- 527	Accept.
C2	1040.	967	848- 1080	877- 1050	Accept.

Performance Evaluation Report
USEPA Water Pollution Study WP034

Date: 29AUG95

Participant ID: KSCC9C2 Type: OTHER Requesting Office: RS

Sample Number	Reported Value	True Value*	Acceptance Limits	Warning Limits	Performance Evaluation
6-ANTIMONY					
C3	130.	127	78.1- 151	87.2- 142	Accept.
C4	430.	441	287- 535	318- 504	Accept.
7-SILVER					
C3	35.7	36.9	31.2- 42.7	32.6- 41.3	Accept.
C4	248.	260	225- 296	234- 287	Accept.
8-THALLIUM					
C3	240.	231	183- 270	194- 259	Accept.
C4	810.	891	708- 1040	750- 997	Accept.
4-MOLYBDENUM					
C3	28.1	31.0	24- 37.7	25.8- 35.9	Accept.
C4	248.	250	202- 295	215- 283	Accept.
5-STRONTIUM					
C3	45.2	45.3	36.7- 52.5	38.8- 50.4	Accept.
C4	289.	290	243- 334	255- 323	Accept.
6-TITANIUM					
C3	38.0	38.0	29.6- 46.7	31.9- 44.4	Accept.
C4	147.	142	121- 161	126- 156	Accept.
MINERALS IN MILLIGRAMS/LITER (EXCEPT AS NOTED)					
9-PH-UNITS					
C3	7.62	7.60	7.4- 7.76	7.44- 7.72	Accept.
C4	9.05	9.00	8.61- 9.35	8.7- 9.26	Accept.
0-SPEC. COND. (UMHOS/CM AT 25 C)					
C1	425.	414	390- 450	397- 442	Accept.
C2	870.	859	805- 935	821- 918	Accept.
1-TDS AT 180 C					
C1	228.	237	172- 304	189- 288	Accept.
2-TOTAL HARDNESS (AS CACO3)					
C1	82.3	78.4	70.3- 87.3	72.4- 85.2	Accept.
C2	325.	314	286- 340	293- 334	Accept.
3-CALCIUM					
C1	22.1	21.0	18.5- 23.6	19.1- 22.9	Accept.
C2	82.4	78.0	69.3- 86.7	71.5- 84.5	Accept.
4-MAGNESIUM					
C1	6.45	6.30	5.52- 7.16	5.73- 6.95	Accept.
C2	30.8	29.0	25.3- 32.9	26.3- 32	Accept.
5-SODIUM					
C1	30.5	29.5	26.7- 32.6	27.4- 31.5	Accept.
C2	18.2	18.1	16.2- 20.3	16.7- 19.8	Accept.
6-POTASSIUM					
C1	33.8	32.0	28- 36.1	29- 35.1	Accept.
C2	10.9	10.2	8.73- 11.7	9.1- 11.3	Accept.
7-TOTAL ALKALINITY (AS CACO3)					
C1	45.5	45.0	39.7- 51.1	41.1- 49.6	Accept.
C2	10.2	10.0	7.41- 14.2	8.26- 13.3	Accept.

Performance Evaluation Report
USEPA Water Pollution Study WP034

Date: 29AUG95

Participant ID: KSCC9C2 Type: OTHER Requesting Office: RS

Sample Number	Reported Value	True Value*	Acceptance Limits	Warning Limits	Performance Evaluation
NUTRIENTS IN MILLIGRAMS/LITER					
1-AMMONIA-NITROGEN					
C1	8.22	8.80	7.05- 10.4	7.45- 9.99	Accept.
C2	4.00	3.90	3.07- 4.7	3.26- 4.5	Accept.
2-NITRATE-NITROGEN					
C1	5.66	6.02	4.85- 7.06	5.12- 6.8	Accept.
02	22.0	23.1	18.6- 27	19.6- 26	Accept.
DEMANDS IN MILLIGRAMS/LITER					
6-COD					
01	50.5	48.1	33.4- 55.6	36.2- 52.8	Accept.
C2	19.2	15.9	6.15- 23.6	8.35- 21.4	Accept.
7-TOC					
C1	19.3	19.0	16.1- 22	16.9- 21.3	Accept.
02	6.50	6.30	5.16- 7.46	5.45- 7.16	Accept.
8-5-DAY BOD					
C1	30.0	30.2	17.3- 42.3	20.5- 39.2	Accept.
02	7.22	9.99	4.82- 15.4	6.14- 14	Accept.
PCB'S IN MICROGRAMS/LITER					
0-PCB-AROCLOR 1016/1242					
02	4.92	6.74	1.62- 9.74	2.65- 8.71	Accept.
6-PCB-ARCCLOR 1260					
C1	1.59	1.61	0.694- 2.11	0.872- 1.93	Accept.
PCB'S IN OIL IN MILLIGRAMS/KILOGRAM					
0-PCB IN OIL- 1254					
02	26.4	22.7	2.97- 37	7.33- 32.7	Accept.
1-PCB IN OIL- 1260					
01	45.4	42.7	3.16- 62.9	10.8- 55.2	Accept.
PESTICIDES IN MICROGRAMS/LITER					
7-ALDRIN					
C1	1.30	1.63	0.186- 2.49	0.475- 2.2	Accept.
C2	0.390	0.364	0.0925-0.506	0.144-0.454	Accept.
8-DIELDRIN					
C1	2.60	4.20	2.05- 5.91	2.53- 5.42	Accept.
C2	1.20	1.57	0.743- 2.28	0.936- 2.09	Accept.
9-DDD					
C1	3.20	3.88	2.15- 5.17	2.54- 4.79	Accept.
02	0.90	1.14	0.573- 1.62	0.704- 1.49	Accept.
0-DDE					
01	2.22	2.71	1.33- 3.54	1.61- 3.26	Accept.
C2	1.61	1.07	0.543- 1.41	0.653- 1.3	Not Accept.
1-DDT					
C1	2.10	2.84	1.4- 3.84	1.71- 3.53	Accept.
02	1.50	1.42	0.68- 1.87	0.829- 1.72	Accept.

Performance Evaluation Report
USEPA Water Pollution Study WP034

Date: 29AUG95

rticipant ID: KS00902 Type: OTHER Requesting Office: KS

Sample Number	Reported Value	True Value*	Acceptance Limits	Warning Limits	Performance Evaluation
2-HEPTACHLOR					
C1	1.90	2.23	0.449- 3.24	0.799- 2.89	Accept.
C2	0.29	0.426	0.0989- 0.6	0.162-0.537	Accept.
3-CHLORDANE					
C3	1.71	1.89	1- 2.57	1.2- 2.37	Accept.
C4	3.38	4.36	1.91- 6.46	2.48- 5.88	Accept.
8-HEPTACHLOR EPOXIDE					
C1	1.40	1.93	1.05- 2.53	1.23- 2.34	Accept.
C2	0.303	0.386	0.206-0.507	0.244-0.469	Accept.
VOLATILE HALOCARBONS IN MICROGRAMS/LITER					
4-1,2 DICHLOROETHANE					
C1	65.0	63.4	44.9- 81.9	49.6- 77.2	Accept.
C2	14.4	13.7	9.36- 19.1	10.6- 17.9	Accept.
5-CHLOROFORM					
C1	60.9	58.3	40.4- 74.9	44.7- 70.6	Accept.
C2	17.9	17.8	12.8- 23.1	14.1- 21.8	Accept.
6-1,1,1 TRICHLOROETHANE					
C1	48.7	48.1	31.6- 62.2	35.4- 58.3	Accept.
C2	16.8	18.6	12.5- 23.8	14- 22.4	Accept.
7-TRICHLOROETHENE					
C1	56.1	57.4	36.7- 75.4	41.5- 70.5	Accept.
C2	12.1	13.3	8.82- 17.2	9.87- 16.2	Accept.
8-CARBONTETRACHLORIDE					
C1	64.1	64.2	40.7- 89.4	46.8- 83.2	Accept.
C2	15.7	17.0	11.7- 23	13.1- 21.6	Accept.
9-TETRACHLOROETHENE					
C1	53.0	54.2	36.7- 68.4	40.7- 64.4	Accept.
C2	15.1	16.6	11.7- 21	12.9- 19.8	Accept.
0-BROMODICHLOROMETHANE					
C1	66.0	65.4	46.9- 85.7	51.8- 80.7	Accept.
C2	17.2	18.5	13.2- 24.2	14.6- 22.8	Accept.
1-DIBROMOCHLOROMETHANE					
C1	52.6	53.7	36.7- 70.5	40.9- 66.3	Accept.
C2	9.51	12.8	8.69- 16.5	9.67- 15.5	Ck. for Err.
2-BROMOFORM					
C1	53.2	55.0	36- 74.7	40.9- 69.8	Accept.
C2	11.0	15.0	8.95- 20.3	10.4- 18.8	Accept.
3-METHYLENE CHLORIDE					
C1	62.0	58.1	35.4- 81.7	41.2- 75.9	Accept.
C2	14.7	12.3	7.72- 17.6	8.96- 16.3	Accept.
4-CHLOROBENZENE					
C1	54.9	54.7	40.3- 67.2	43.7- 63.8	Accept.
C2	14.6	15.2	11.6- 18.6	12.5- 17.7	Accept.
VOLATILE AROMATICS IN MICROGRAMS/LITER					
5-BENZENE					
C1	12.0	11.4	7.74- 14.9	8.63- 14	Accept.
C2	48.0	49.1	35.3- 63.6	38.9- 60.1	Accept.

KANSAS
DEPARTMENT OF HEALTH & ENVIRONMENT
BILL GRAVES, GOVERNOR
Gary R. Mitchell, Secretary

M E M O R A N D U M

To: Participating Laboratories

From: Jack R. McKenzie and Aurora P. Shields
Laboratory Improvement Specialists

Date: May 29, 1998

Re: WP039 Data Evaluation

Enclosed please find an evaluation of the data which your laboratory reported for EPA Performance Evaluation (P.E.) Study WP039. Review this data carefully. Any data which has been evaluated as other than acceptable should be closely checked and the procedures used to obtain that value scrutinized carefully. The quality of the data which your laboratory is routinely producing for such a parameter may be in question.

Please respond in writing to our office **within 15 days of receipt of this report** concerning any corrective action your laboratory has taken on unacceptable parameters. This information may be considered when our office evaluates your parameters for future certification.

If you have any questions please feel free to contact our office at (785) 296-1639 - Jack McKenzie or (785) 296-6198 - Aurora Shields.

Division of Health & Environmental Laboratories
Forbes Field, Bldg. 740, Topeka, KS 66620-0001
http://www.state.ks.us/public/kdhe/lab.html

Telephone: Jack McKenzie (785) 296-1639
Aurora Shields (785) 296-6198
FAX (785) 296-8068

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Performance Evaluation Report
USEPA Water Pollution Study WP039

Participant ID: KS00902 Type: OTHER Requesting Office: KS

Sample Number	Reported Value	True Value*	Acceptance Limits	Warning Limits	Performance Evaluation
TRACE METALS IN MICROGRAMS/LITER					
001-ALUMINUM					
01	555	489	420- 562	437- 544	Ck. for Err.
002-ARSENIC					
01	778	864	739- 1010	772- 972	Accept.
003-BERYLLIUM					
02	183	191	170- 214	176- 208	Accept.
004-CADMIUM					
01	40.3	33.9	28.7- 39.1	30- 37.8	Not Accept.
005-COBALT					
01	86.6	82.3	71.5- 92.4	74.1- 89.7	Accept.
006-CHROMIUM					
01	224	216	189- 244	196- 237	Accept.
007-COPPER					
01	80.6	74.3	68.2- 84.3	70.2- 82.3	Accept.
008-IRON					
01	206	191	165- 218	171- 211	Accept.
009-MERCURY					
01	3.74	0.563	0.307-0.782	0.366-0.722	Not Accept.
010-MANGANESE					
01	553	530	485- 587	498- 574	Accept.
011-NICKEL					
01	795	702	674- 780	688- 767	Not Accept.
012-LEAD					
01	2030	1900	1690- 2110	1740- 2060	Accept.
013-SELENIUM					
01	773	760	625- 862	655- 833	Accept.
014-VANADIUM					
01	1520	1420	1320- 1560	1350- 1530	Accept.
015-ZINC					
01	138	131	113- 150	118- 146	Accept.
016-ANTIMONY					
02	261	277	198- 325	214- 309	Accept.
017-SILVER					
02	227	130	118- 150	122- 146	Not Accept.
018-THALLIUM					
02	58.0	141	121- 167	127- 161	Not Accept.
074-MOLYBDENUM					
02	588	589	521- 662	539- 644	Accept.
MINERALS IN MILLIGRAMS/LITER (EXCEPT AS NOTED)					
020-SPEC. COND.(UMHOS/CM AT 25 C)					
01	289	296	271- 313	276- 308	Accept.
021-TDS AT 180 C					
01	133	156	127- 200	136- 191	Ck. for Err.
022-TOTAL HARDNESS(AS CACO3)					
01	16.1	14.8	11.7- 18.8	12.6- 17.9	Accept.

Performance Evaluation Report
USEPA Water Pollution Study WP039

Participant ID: KS00902 Type: OTHER Requesting Office: KS

Sample Number	Reported Value	True Value*	Acceptance Limits	Warning Limits	Performance Evaluation
023-CALCIUM					
01	2.85	2.51	1.98- 3.23	2.14- 3.07	Accept.
024-MAGNESIUM					
01	2.19	2.08	1.84- 2.37	1.91- 2.31	Accept.
025-SODIUM					
01	32.1	31.8	28- 36.2	29- 35.1	Accept.
026-POTASSIUM					
01	30.4	30.0	24.4- 35.3	25.7- 33.9	Accept.
027-TOTAL ALKALINITY(AS CACO3)					
01	39.1	36.8	32.3- 44.7	33.8- 43.1	Accept.
028-CHLORIDE					
01	9.93	10.8	8.3- 12.7	8.85- 12.2	Accept.
030-SULFATE					
01	54.7	58.0	49- 67.3	51.3- 65	Accept.
NUTRIENTS IN MILLIGRAMS/LITER					
031-AMMONIA-NITROGEN					
01	0.79	0.840	0.594- 1.13	0.658- 1.07	Accept.
032-NITRATE-NITROGEN					
01	1.11	1.10	0.868- 1.31	0.921- 1.26	Accept.
033-ORTHOPHOSPHATE					
01	0.25	0.250	0.203-0.296	0.214-0.285	Accept.
034-KJELDAHL-NITROGEN					
02	0.76	0.360	D.L. -0.907	0.0785-0.794	Accept.
035-TOTAL PHOSPHORUS					
02	0.20	0.170	0.15-0.252	0.162- 0.24	Accept.
DEMANDS IN MILLIGRAMS/LITER					
036-COD					
01	180	192	139- 232	151- 220	Accept.
038-5-DAY BOD					
01	111	119	63.5- 174	77.4- 160	Accept.
PCB'S IN MICROGRAMS/LITER					
040-PCB-AROCLOR 1016/1242					
02	7.9	6.40	2.55- 8.54	3.3- 7.78	Ck. for Err.
045-PCB-AROCLOR 1254					
01	3.2	3.29	1.45- 4.34	1.81- 3.98	Accept.
PCB'S IN OIL IN MILLIGRAMS/KILOGRAM					
099-PCB IN OIL- 1016/1242					
02	40.6	41.7	6.01- 57.7	12.6- 51.1	Accept.
101-PCB IN OIL- 1260					
01	12.9	16.6	2.93- 23.7	5.57- 21	Accept.
PESTICIDES IN MICROGRAMS/LITER					
047-ALDRIN					
01	1.03	1.48	0.354- 2.12	0.576- 1.89	Accept.

Performance Evaluation Report
USEPA Water Pollution Study WP039

Participant ID: KS00902 Type: OTHER Requesting Office: KS

Sample Number	Reported Value	True Value*	Acceptance Limits	Warning Limits	Performance Evaluation
048-DIELDRIN					
01	1.42	1.72	0.949- 2.14	1.1- 1.99	Accept.
049-DDD					
01	1.54	2.35	1.19- 2.94	1.41- 2.72	Accept.
050-DDE					
01	1.30	1.47	0.791- 2.56	1.01- 2.34	Accept.
051-DDT					
01	2.17	3.25	1.07- 3.76	1.41- 3.43	Accept.
052-HEPTACHLOR					
01	0.5	0.764	0.236- 1.03	0.335-0.927	Accept.
053-CHLORDANE					
02	12.0	12.7	5.86- 18.9	7.5- 17.3	Accept.
078-HEPTACHLOR EPOXIDE					
01	0.36	0.586	0.317-0.693	0.364-0.646	Ck. for Err.
VOLATILE HALOCARBONS IN MICROGRAMS/LITER					
054-1,2 DICHLOROETHANE					
01	22.2	22.8	15.8- 28.9	17.5- 27.3	Accept.
055-CHLOROFORM					
01	24.8	27.2	18.5- 33.3	20.3- 31.5	Accept.
056-1,1,1 TRICHLOROETHANE					
01	13.7	17.4	11.3- 23.3	12.8- 21.8	Accept.
057-TRICHLOROETHENE					
01	10.5	12.7	7.88- 15.3	8.81- 14.4	Accept.
058-CARBONTETRACHLORIDE					
01	12.0	15.2	8.37- 20.9	9.94- 19.3	Accept.
059-TETRACHLOROETHENE					
01	14.3	17.5	11.1- 22.3	12.5- 20.9	Accept.
060-BROMODICHLOROMETHANE					
01	18.5	21.6	15.7- 25.8	17- 24.5	Accept.
061-DIBROMOCHLOROMETHANE					
01	14.1	20.4	13.7- 25	15.1- 23.6	Ck. for Err.
062-BROMOFORM					
01	26.2	36.8	21.7- 47.5	24.9- 44.3	Accept.
063-METHYLENE CHLORIDE					
01	30.2	33.6	21.8- 41.8	24.3- 39.3	Accept.
064-CHLOROBENZENE					
01	15.4	16.6	11.8- 20.6	12.9- 19.5	Accept.
VOLATILE AROMATICS IN MICROGRAMS/LITER					
065-BENZENE					
01	45.0	46.4	35- 57.9	37.9- 55	Accept.
066-ETHYLBENZENE					
01	25.8	27.3	19.2- 34.5	21.1- 32.6	Accept.
067-TOLUENE					
01	65.5	62.6	46.5- 76.5	50.3- 72.8	Accept.
094-1,2-DICHLOROBENZENE					
01	12.6	13.8	9.42- 16.2	10.3- 15.3	Accept.

Performance Evaluation Report
USEPA Water Pollution Study WP039

Participant ID: KS00902 Type: OTHER Requesting Office: KS

Sample Number	Reported Value	True Value*	Acceptance Limits	Warning Limits	Performance Evaluation
095-1,4-DICHLOROBENZENE					
01	17.4	18.8	13.6- 23.9	14.9- 22.6	Accept.
096-1,3-DICHLOROBENZENE					
01	19.7	22.4	15.7- 26.4	17.1- 25.1	Accept.
MISCELLANEOUS ANALYTES					
019-PH-UNITS					
01	5.07	5.03	4.93- 5.14	4.95- 5.12	Accept.
071-TOTAL CYANIDE(IN MG/L)					
01	0.7	0.639	0.447-0.836	0.496-0.787	Accept.
072-NON-FILTERABLE RESIDUE(IN MG/L)					
01	66	96.0	36.8- 109	45.7- 100	Accept.
097-TOTAL PHENOLICS(IN MG/L)					
01	0.63	0.799	0.443- 1.15	0.536- 1.06	Accept.
098-TOTAL RESIDUAL CHLORINE(IN MG/L)					
01	0.282	0.280	0.229-0.439	0.256-0.412	Accept.
104-OIL AND GREASE (HEXANE EXTRACTION)(IN MG/L)					
01	33.1	44.0	24.2- 58	28.7- 53.5	Accept.

********** END OF DATA FOR KS00902 **********
NOTE: FOR LIMITS AND TRUE VALUES, ASSUME THREE SIGNIFICANT DIGITS.
********** END OF REPORT FOR KS00902 **********

* Based on gravimetric calculations, or a reference value when necessary.

Recd. 12/10/98
~~Mail~~ 12

KANSAS
DEPARTMENT OF HEALTH & ENVIRONMENT
BILL GRAVES, GOVERNOR
Gary R. Mitchell, Secretary

MEMORANDUM

To: Participating Laboratories

From: Jack R. McKenzie and Aurora P. Shields
Laboratory Improvement Specialists

Date: December 8, 1998

Re: WP040 Data Evaluation

Enclosed please find an evaluation of the data which your laboratory reported for EPA Performance Evaluation (P.E.) Study WP040. Review this data carefully. Any data which has been evaluated as other than acceptable should be closely checked and the procedures used to obtain that value scrutinized carefully. The quality of the data which your laboratory is routinely producing for such a parameter may be in question.

Please respond in writing to our office **within 15 days of receipt of this report** concerning any corrective action your laboratory has taken on unacceptable parameters. This information may be considered when our office evaluates your parameters for future certification.

Enclosed you will find a two page document (front & back) provided by EPA which contains information on the privatization of the PE studies and a PE Guidance letter from our office.

If you have any questions please feel free to contact our office at (785) 296-1639 - Jack McKenzie or (785) 296-6198 - Aurora Shields.

Division of Health & Environmental Laboratories
Forbes Field, Bldg. 740, Topeka, KS 66620-0001
http://www.kdhe.state.ks.us/lipo

Telephone: Jack McKenzie (785) 296-1639
Aurora Shields (785) 296-6198
FAX (785) 296-8068

Committed to Excellence in Analytical Laboratory Science

Information on the Privatization of the Performance Evaluation Studies

Background:

The Environmental Protection Agency (US EPA), after consultation with stakeholders regarding various options, decided (FR, Volume 62, No. 113, Thursday, June 12, 1997) to externalize components of the laboratory performance evaluation (PE) studies programs that the Agency has conducted to assess the proficiency of laboratories testing drinking water and wastewater to entities outside EPA. Under the externalized program, EPA would issue standards for the operation of the program, the National Institute of Standards and Technology (NIST) would develop a program to accredit Proficiency Testing (PT) Providers, the PT Providers would develop and manufacture PT materials and conduct PT studies, environmental laboratories would participate in these externalized PT studies by purchasing PT samples directly from PT Providers, and Certifying Authorities would be responsible for certifying environmental laboratories performing sample analyses in support of the various water programs administered by the States and EPA under the Clean Water Act and the Safe Drinking Water Act.

What are the major changes between the EPA provided Performance Evaluation Studies Program and the privatized Proficiency Testing Program?

- Under the EPA PE program, all samples were provided by one source (an EPA contractor) to the laboratories at no cost to the lab. Under the privatized PT program, the laboratory will purchase samples from NIST accredited providers. Unless otherwise directed by the States, the laboratory may choose any NIST accredited provider. If no NIST accredited providers are available, any commercial source of PT samples is acceptable.

- The acceptance criteria used in evaluation of each laboratory's data under the EPA PE program was either fixed limits set by regulations or statistically derived limits based on the study data. Under the privatized PT program, all providers will use either the fixed limits set by regulations or statistically derived limits based on historical EPA PE studies.

- Under the EPA PE studies, two studies per year were available for water supply chemistry, water supply microbiology and water pollution chemistry studies. DMRQA chemistry and toxicity studies were offered once a year while water supply radiochemistry studies were offer two or three times a year depending on the radionuclide. Laboratories will be required to successfully participate in the number of PT studies prescribed by the regulatory authority overseeing the particular program for which the laboratory is seeking certification. If a laboratory is seeking certification under the Safe Drinking Water Act, the minimum Federal requirement is participation in a PT study once a year. The States have the option to increase the number of participations per year. The States also determine the frequency of participation in WP studies. EPA's Office of Enforcement and Compliance Assurance (OECA) will issue letters to permittees informing them that they

KANSAS
DEPARTMENT OF HEALTH & ENVIRONMENT
BILL GRAVES, GOVERNOR
Gary R. Mitchell, Secretary

The privatization of the Performance Evaluation studies (PE) by EPA will create several changes in the way the studies are handled by the laboratories. In our letter from December 2, 1998 we indicated you must contact a PE vendor of your choice and order the PE studies for the time scheduled indicated in the letter.

This letter is to provide you with additional guidelines:

1. A list of PE vendors is attached. Please note this list does not mean EPA or Kansas approval or endorsement of these vendors. It simply means these vendors are currently selling PE studies.

2. In our letter of December 2, 1998 we indicated the laboratory will have 30 days to complete each study. Please note this has been changed to 45 days from the scheduled study shipment date. The schedule for the PE studies is as follows:

Schedule for PE studies	**NOTE**
The laboratory contacts the PE provider to order WS and/or WP studies for the year.	1. Indicate which studies you need (WS and/or WP) 2. Give PE provider your EPA PE number (KS#####) 3. Indicate which contaminants you need 4. Indicate the required schedule time for the studies: WS are scheduled for February and August WP are scheduled for April and October
Provider ships WS samples to the participating laboratories in February and August. Provider ships WP samples to participating laboratories in April and October.	Failure to participate in the required time schedule is considered a failed study.

Division of Health and Environmental Laboratories
Forbes Field, Bldg. 740, Topeka, KS 66620-0001
http://www.kdhe.state.ks.us/lipo

Committed to Excellence in Analytical Laboratory Science

Telephone: Jack McKenzie (785) 296-1639
Aurora Shields (785) 296-6198
FAX (785) 296-8068

LIST OF PE PROVIDERS

ENVIRONMENTAL RESOURCE ASSOCIATES	303-431-8454 1-800-ERA-0122
ACCUDSTANDARD INC.	1-800-442-5290
ULTRA SCIENTIFIC	1-800-338-1754
NSI ENVIRONMENTAL SOLUTIONS, INC.	1-800-234-7837
ANALYTICAL PRODUCTS GROUP	1-800-272-4442

Performance Evaluation Report
USEPA Water Pollution Study WP040

Participant ID: KS00902 Type: OTHER Requesting Office: KS

Sample Number	Reported Value	True Value*	Acceptance Limits	Warning Limits	Performance Evaluation
TRACE METALS IN MICROGRAMS/LITER					
001-ALUMINUM					
01	2810	3105	2740- 3440	2830- 3350	Ck. for Err.
002-ARSENIC					
01	180	160	128- 190	136- 183	Accept.
003-BERYLLIUM					
02	35.7	36.9	33.1- 42.2	34.2- 41	Accept.
004-CADMIUM					
01	164	170	147- 193	152- 187	Accept.
005-COBALT					
01	497	503	449- 561	464- 547	Accept.
006-CHROMIUM					
01	632	650	579- 726	598- 708	Accept.
007-COPPER					
01	693	700	654- 781	670- 765	Accept.
008-IRON					
01	797	834	744- 936	768- 912	Accept.
009-MERCURY					
01	1.45	1.15	0.813- 1.47	0.895- 1.39	Ck. for Err.
010-MANGANESE					
01	242	240	216- 259	222- 254	Accept.
011-NICKEL					
01	2640	2501	2340- 2860	2400- 2790	Accept.
012-LEAD					
01	60.4	70.6	61.7- 83.3	64.4- 80.6	Not Accept.
013-SELENIUM					
01	257	260	189- 297	202- 284	Accept.
014-VANADIUM					
01	4250	4202	3880- 4640	3980- 4550	Accept.
015-ZINC					
01	682	631	563- 709	581- 690	Accept.
016-ANTIMONY					
02	505	499	381- 590	407- 564	Accept.
017-SILVER					
02	820	851	736- 930	760- 906	Accept.
018-THALLIUM					
02	872	841	747- 970	775- 942	Accept.
074-MOLYBDENUM					
02	17	18.2	14- 22	15- 21	Accept.
MINERALS IN MILLIGRAMS/LITER (EXCEPT AS NOTED)					
020-SPEC. COND.(UMHOS/CM AT 25 C)					
01	492	525	471- 544	480- 535	Accept.
021-TDS AT 180 C					
01	264	274	230- 337	243- 323	Accept.
022-TOTAL HARDNESS(AS CACO3)					
01	116	105	93.3- 119	96.5- 116	Accept.

Performance Evaluation Report
USEPA Water Pollution Study WP040

Participant ID: KS00902 Type: OTHER Requesting Office: KS

Sample Number	Reported Value	True Value*	Acceptance Limits	Warning Limits	Performance Evaluation
023-CALCIUM					
01	28.4	29.0	25.1- 33.2	26.1- 32.2	Accept.
024-MAGNESIUM					
01	7.99	8.00	7.06- 8.86	7.29- 8.64	Accept.
025-SODIUM					
01	42.3	39.0	35.7- 44.1	36.7- 43	Accept.
026-POTASSIUM					
01	26.3	25.0	21.4- 28.8	22.4- 27.9	Accept.
027-TOTAL ALKALINITY(AS CACO3)					
01	40.6	42.4	35.5- 48.1	37.1- 46.5	Accept.
028-CHLORIDE					
01	80.4	74.9	68.1- 83.2	70- 81.3	Accept.
030-SULFATE					
01	80.6	69.0	58.3- 78.4	60.8- 75.9	Not Accept.
NUTRIENTS IN MILLIGRAMS/LITER					
031-AMMONIA-NITROGEN					
01	5.29	4.80	3.89- 5.84	4.13- 5.6	Accept.
032-NITRATE-NITROGEN					
01	12.5	12.0	10.1- 13.9	10.6- 13.4	Accept.
033-ORTHOPHOSPHATE					
01	0.56	0.580	0.496-0.669	0.516-0.648	Accept.
035-TOTAL PHOSPHORUS					
02	4.11	4.00	3.3- 4.68	3.47- 4.52	Accept.
DEMANDS IN MILLIGRAMS/LITER					
036-COD					
01	66.6	60.7	42.5- 73.5	46.5- 69.5	Accept.
038-5-DAY BOD					
01	45.5	37.6	17.7- 57.2	22.6- 52.2	Accept.
PCB'S IN MICROGRAMS/LITER					
042-PCB-AROCLOR 1232					
01	3.00	4.73	1.1- 7.18	1.88- 6.41	Accept.
046-PCB-AROCLOR 1260					
02	3.09	3.24	1.29- 4.6	1.71- 4.18	Accept.
PCB'S IN OIL IN MILLIGRAMS/KILOGRAM					
099-PCB IN OIL- 1016/1242					
02	40.4	38.7	3.96- 57.2	10.8- 50.3	Accept.
100-PCB IN OIL- 1254					
01	23.4	24.0	D.L. - 40.2	3.97- 34.9	Accept.
PESTICIDES IN MICROGRAMS/LITER					
047-ALDRIN					
01	0.81	0.940	0.164- 1.45	0.326- 1.29	Accept.
048-DIELDRIN					
01	4.54	2.78	1.55- 3.96	1.86- 3.66	Not Accept.

Performance Evaluation Report
USEPA Water Pollution Study WP040

Participant ID: KS00902 Type: OTHER Requesting Office: KS

	Sample Number	Reported Value	True Value*	Acceptance Limits	Warning Limits	Performance Evaluation
049-DDD	01	7.40	5.25	3.21- 7.12	3.7- 6.62	Not Accept.
050-DDE	01	3.02	3.64	1.74- 5.1	2.16- 4.68	Accept.
051-DDT	01	6.80	8.42	2.61- 12.7	3.89- 11.5	Accept.
052-HEPTACHLOR	01	2.13	2.49	0.837- 3.41	1.16- 3.08	Accept.
053-CHLORDANE	02	4.10	4.81	2.08- 6.8	2.68- 6.2	Accept.
078-HEPTACHLOR EPOXIDE	01	2.12	1.98	1.13- 2.42	1.29- 2.26	Accept.
VOLATILE HALOCARBONS IN MICROGRAMS/LITER						
054-1,2 DICHLOROETHANE	01	14.5	14.6	9.85- 18.5	10.9- 17.4	Accept.
055-CHLOROFORM	01	17.6	18.4	13.7- 21.7	14.7- 20.7	Accept.
056-1,1,1 TRICHLOROETHANE	01	31.7	32.8	22.2- 41.1	24.6- 38.7	Accept.
057-TRICHLOROETHENE	01	18.4	23.4	15.6- 29.8	17.4- 28	Accept.
058-CARBONTETRACHLORIDE	01	27.2	26.3	16.3- 32.9	18.3- 30.8	Accept.
059-TETRACHLOROETHENE	01	29.7	32.5	21.6- 39.5	23.8- 37.2	Accept.
060-BROMODICHLOROMETHANE	01	15.2	16.5	10.9- 19.8	12- 18.7	Accept.
061-DIBROMOCHLOROMETHANE	01	33.2	32.7	21.4- 40.6	23.8- 38.2	Accept.
062-BROMOFORM	01	15.0	14.7	8.12- 18.7	9.44- 17.3	Accept.
063-METHYLENE CHLORIDE	01	46.3	44.1	29.7- 58.8	33.3- 55.1	Accept.
064-CHLOROBENZENE	01	23.2	24.7	17.6- 29.3	19- 27.8	Accept.
VOLATILE AROMATICS IN MICROGRAMS/LITER						
065-BENZENE	01	26.1	25.7	21.9- 33.7	23.4- 32.2	Accept.
066-ETHYLBENZENE	01	40.2	42.6	29.9- 54	32.9- 50.9	Accept.
067-TOLUENE	01	30.8	32.3	23.9- 39.8	25.9- 37.8	Accept.
094-1,2-DICHLOROBENZENE	01	32.3	36.3	27.1- 45.1	29.4- 42.8	Accept.
095-1,4-DICHLOROBENZENE	01	43.5	41.6	30.4- 52.3	33.1- 49.6	Accept.

Performance Evaluation Report
USEPA Water Pollution Study WP040

Participant ID: KS00902 Type: OTHER Requesting Office: KS

Sample Number	Reported Value	True Value*	Acceptance Limits	Warning Limits	Performance Evaluation
096-1,3-DICHLOROBENZENE					
01	28.5	33.7	23.7- 40.4	25.8- 38.3	Accept.
MISCELLANEOUS ANALYTES					
019-PH-UNITS					
01	8.44	8.60	8.31- 8.92	8.38- 8.84	Accept.
071-TOTAL CYANIDE(IN MG/L)					
01	0.16	0.140	0.089-0.184	0.101-0.172	Accept.
072-NON-FILTERABLE RESIDUE(IN MG/L)					
01	47	64.0	12.4- 80.7	20.9- 72.1	Accept.
097-TOTAL PHENOLICS(IN MG/L)					
01	0.058	0.0668	0.0261-0.108	0.0367-0.097	Accept.
098-TOTAL RESIDUAL CHLORINE(IN MG/L)					
01	1.19	0.930	0.811- 1.32	0.878- 1.25	Accept.
104-OIL AND GREASE (HEXANE EXTRACTION)(IN MG/L)					
01	19.9	19.1	5.65- 28.7	8.82- 25.6	Accept.

********** END OF DATA FOR KS00902 **********
NOTE: FOR LIMITS AND TRUE VALUES, ASSUME THREE SIGNIFICANT DIGITS.
********** END OF REPORT FOR KS00902 **********

* Based on gravimetric calculations, or a reference value when necessary.

Analytical Products Group, Inc.

PERFORMANCE REPORT

April 1999

Prepared for

Analytical Management Laboratory

15130 South Keeler Suite B
Olathe, KS 66062

Laboratory Code 3655

Laboratory of
EXCELLENCE

WP Performance Evaluation Report Introduction and Explanation of Terms

Introduction:
The APG Proficiency Testing Program has been designed to evaluate your laboratory's performance against other participating laboratories on the same set of standards. Sample levels have been chosen to assist you in ensuring that the procedure is in-control. In most cases, sample levels are not designed to challenge the detection limits of the method.

The following information is provided to assist you in the interpretation of the report. All statistical measures have been calculated based upon generally accepted formulae.

Your Result: Every effort has been made to insure correct data entry. Should you find a data entry error please notify Analytical Products Group and we will adjust the data and reissue the report.

Assigned Value: The reported Assigned Value is the calculated True Value of the standard based upon the actual composition of the standard.

Certified Value: Verification of the Assigned Value based upon analysis by APG versus a NIST SRM or an external standard if a NIST SRM is not available. The reported Uncertainty is expressed as $2s/\sqrt{n}$.

EPA Mean: This value is the Mean value calculated as required by the US EPA National Standards for Water Proficiency Testing Program or is the actual Mean for non-EPA parameters

EPA Standard Deviation: This parameter is also calculated based upon the National Standards for Water Proficiency Testing Program or is the actual Standard Deviation for non-EPA parameters

Solids Standards: Solids standards are individually prepared and each standard has a unique Assigned Value. The EPA Mean and Standard Deviation are calculated on the appropriate Assigned Value.

Deviation from the Mean or z score: This result is an expression of how far your reported value was from the Mean of all reporting laboratories in terms of number of Standard Deviations. As a point of reference, EPA Warning Limits are 2 deviations from the Mean and EPA Acceptance Limits are 3 deviations from the Mean.

Evaluation Line: Following the Deviation from the Mean for each parameter is an Evaluation line. The Evaluation line may have one of five entries:

Entry	Description
Unusable	A less than or text value was reported which could not be evaluated.
Check for Error	Data between 2 and 3 Deviations from the Mean. Data outside the 95% Confidence Interval but within EPA Acceptance Limits.
Unacceptable	Data more than 3 Deviations from the Mean. Data outside the 99% Confidence Interval, outside the EPA Acceptance Limits.
Blank	No data was reported for this parameter.
Acceptable	Data within acceptable limits.

These notations can be used as a guideline in evaluating your performance.

Comment Section: This section contains comments submitted to Analytical Products Group by program participants. It also contains comments from APG to participants concerning unusual characteristics of the data set or problems with standards which could influence the interpretation of the data.

Performance Evaluation Summary: This section summarizes performance data in a format similar to the USEPA DMR-QA Report. Detailed information on each parameter is included in the main report.

Certification: This program meets the requirements of the ISO/REMCO Harmonized Protocol for Proficiency Testing of Analytical Laboratories as well as the requirements of ASTM E1301. The statistical techniques used to evaluate the data are consistent with ISO and ASTM guidelines and the National Standards for Water Proficiency Testing Programs. APG is an ISO9001 Certified provider of Proficiency Testing Programs.

Introduction to Laboratory Benchmarking System:

The enclosed report includes a ranking summary of your laboratory's performance in the PT program. Several different measures of performance are provided and each should be interpreted separately. The following sections provide a description of the various ranking methods and their interpretation. These rankings are based upon a broad group of laboratories which analyzed the same samples as your laboratory. Laboratories in the program analyzed the samples as single blind PT samples.

Percent Acceptable Data:

The percent acceptable data is calculated as the percent of actual results reported which were within 99% confidence limits for the parameter. Results are reported for your laboratory and the overall average percent acceptable data achieved by all laboratories.

This is your laboratory's relative rank out of all possible ranks of in the current study. It should be noted that several laboratories can achieve the same rank. The total number of laboratories reporting data for this study is also reported.

Average Percentile: The report includes your laboratory's rank on each parameter and the related percentile rank for that parameter. The average percentile is the average of this data across all parameters reported. It is a different measure of performance than percent acceptable data since it is effected by your relative performance on each parameter. The report provides an average for this measure for all laboratories reporting as well as your laboratory's performance.

Weighted Rank on Percent Acceptable Data:

Because the database includes laboratories of many sizes and capabilities, APG has provided a weighted rank based upon percent acceptable. This measure considers the number of parameters which were run by each laboratory and weights the rank by the total number of parameters evaluated.

Weighted Rank on Average Percentile Data:

This measure also considers the number of parameters run by the laboratory. The weighted rank on percentile data considers not only if the analysis was within the limits but also how close to the Mean the reported result was and the total number of analyses run by the laboratory.

Submitted: June 2, 1999
Thomas V. Coyner
General Manager

Summary of Laboratory Ranking Data
for
Analytical Management Laboratory
Laboratory Code 3655

Summary of National Ranking Data:

Total Number of Laboratories Reporting:	262
Average Percent Acceptable Data:	81.64
Average Percentile for all Parameters:	51.02

Summary of Laboratory Ranking:

Total Data Points Reported:	31
Number of Acceptable Data Points:	31
Percent Acceptable Data:	100.00
Average Percentile for all Parameters:	48.56
Rank on Percent Acceptable Data:	1 of 85
Weighted Rank on Percent Acceptable Data:	51 of 131
Weighted Rank on Average Percentile Data:	59 of 216

Laboratory Code: 3655 Analytical Management Laborator WP Summary for April 1999

Standard: Demand

Parameter	Level	Reported	Assigned	Acceptance Range	z Score	Evaluation
Biochemical Oxygen Demand	WP		69.81	33.367 - 99.208		
Biochemical Oxygen Demand	APG+		177.70	85.413 - 251.463		
Chemical Oxygen Demand	WP		112.40	84.211 - 130.68		
Chemical Oxygen Demand	APG+		286.10	224.978 - 320.534		
Total Organic Carbon	WP		42.31	35.285 - 48.851		
Total Organic Carbon	APG+		107.70	90.082 - 123.578		
Carboneous BOD	WP		59.01	21.639 - 74.954		
Carboneous BOD	APG+		144.60	52.901 - 182.939		

Standard: Acid Extractables

Parameter	Level	Reported	Assigned	Acceptance Range	z Score	Evaluation
4-Chloro-3-methylphenol	WP	22.7	23.51	10.958 - 28.346	1.05	Acceptable
4-Chloro-3-methylphenol	APG+		82.17	50.375 - 86.152		
2-Chlorophenol	WP	24.1	26.49	12.229 - 30.302	0.94	Acceptable
2-Chlorophenol	APG+		102.40	37.42 - 119.81		
2,4-Dimethylphenol	WP	30.7	34.88	14.438 - 37.951	1.14	Acceptable
2,4-Dimethylphenol	APG+		128.80	46.241 - 137.004		
2,4-Dinitrophenol	WP	58.9	68.94	3.443 - 84.754	1.09	Acceptable
2,4-Dinitrophenol	APG+		161.30	16.588 - 196.531		
2-Methyl-4,6-Dinitrophenol	WP	58.7	57.72	35.88 - 68.287	1.22	Acceptable
2-Methyl-4,6-Dinitrophenol	APG+		211.70	90.957 - 266.532		
2-Nitrophenol	WP	12.8	16.02	5.115 - 19.251	0.26	Acceptable
2-Nitrophenol	APG+		61.32	21.73 - 72.404		
4-Nitrophenol	WP	15.5	79.64	0.00 - 106.908	1.89	Acceptable
4-Nitrophenol	APG+		198.80	33.181 - 225.836		
Pentachlorophenol	WP	61.0	60.79	20.269 - 83.894	0.84	Acceptable

Laboratory Code: 3655 Analytical Management Laborator WP Summary for April 1999

Pentachlorophenol	APG+		114.50	80.01 - 135.419		
Phenol	WP	21.6	28.47	4.199 - 32.62	0.67	Acceptable
Phenol	APG+		132.80	12.94 - 156.303		

Standard: Base Neutrals Extractables

Parameter	Level	Reported	Assigned	Acceptance Range	z Score	Evaluation
Butyl benzyl phthalate	WP	36.9	42.61	0.00 - 63.158	0.64	Acceptable
Butyl benzyl phthalate	APG+		199.70	0.00 - 286.258		
Diethyl phthalate	WP	39.8	38.52	6.475 - 52.829	1.31	Acceptable
Diethyl phthalate	APG+		133.40	0.00 - 229.006		
Dimethyl phthalate	WP	80.7	89.76	0.00 - 129.707	1.05	Acceptable
Dimethyl phthalate	APG+		108.90	0.00 - 181.529		
2,6-Dinitrotoluene (2,6-DNT)	WP	35.8	24.24	0.00 - 40.745	2.31	Check for Error
2,6-Dinitrotoluene (2,6-DNT)	APG+		73.41	43.502 - 71.923		
Isophorone	WP	36.6	44.82	24.274 - 51.049	0.23	Acceptable
Isophorone	APG+		70.39	41.426 - 73.58		
Nitrobenzene (NB)	WP	25.6	28.93	13.96 - 33.102	0.64	Acceptable
Nitrobenzene (NB)	APG+		82.63	34.956 - 90.568		
Acenaphthene	WP	24.5	22.82	13.33 - 26.454	2.10	Check for Error
Acenaphthene	APG+		85.48	46.07 - 92.805		
Anthracene	WP	23.1	23.46	15.791 - 25.029	1.74	Acceptable
Anthracene	APG+		62.87	36.052 - 68.508		
Fluoranthene	WP	35.7	37.60	18.477 - 44.345	0.99	Acceptable
Fluoranthene	APG+		112.40	61.22 - 123.061		
Fluorene	WP	38.3	36.09	17.83 - 42.341	2.01	Check for Error
Fluorene	APG+		110.10	57.021 - 121.454		
Naphthalene	WP	22.2	26.42	14.475 - 29.33	0.12	Acceptable
Naphthalene	APG+		85.10	32.517 - 98.085		
Phenanthrene	WP	20.3	23.26	16.889 - 24.03	0.13	Acceptable
Phenanthrene	APG+		55.89	29.153 - 65.224		
Pyrene	WP	25.7	29.27	20.968 - 30.482	0.01	Acceptable
Pyrene	APG+		74.33	28.925 - 94.532		

1,2-Dichlorobenzene	WP	51.1	68.98	22.305 - 81.721	0.09	Acceptable
1,2-Dichlorobenzene	APG+		127.40	36.087 - 151.446		
1,4-Dichlorobenzene	WP	39.2	50.41	15.424 - 56.799	0.44	Acceptable
1,4-Dichlorobenzene	APG+		201.90	43.127 - 204.977		
Hexachlorobenzene	WP	24.8	25.64	4.181 - 38.344	0.62	Acceptable
Hexachlorobenzene	APG+		72.99	19.966 - 98.35		
Hexachlorobutadiene	WP	26.0	31.62	10.364 - 35.113	0.79	Acceptable
Hexachlorobutadiene	APG+		58.25	17.412 - 69.646		
Hexachlorocyclopentadiene	WP	35.3	37.69	0.00 - 48.577	1.44	Acceptable
Hexachlorocyclopentadiene	APG+		98.05	0.00 - 119.61		
Hexachloroethane	WP	17.7	21.74	7.924 - 26.087	0.22	Acceptable
Hexachloroethane	APG+		57.41	16.447 - 68.142		
1,2,4-Trichlorobenzene	WP	24.7	32.16	11.40 - 40.804	0.28	Acceptable
1,2,4-Trichlorobenzene	APG+		104.80	29.196 - 115.11		

Standard: TPH Diesel (DRO)

Parameter	Level	Reported	Assigned	Acceptance Range	z Score	Evaluation
TPH Diesel (DRO)	WP	2.31	0.471	0.449 - 3.306	0.90	Acceptable
TPH Diesel (DRO)	APG+		0.102	0.146 - 0.542		

Standard: TPH Gasoline (GRO)

Parameter	Level	Reported	Assigned	Acceptance Range	z Score	Evaluation
TPH Gasoline (GRO)	WP	0.547	0.429	0.00 - 0.805	1.29	Acceptable
TPH Gasoline (GRO)	APG+		0.782	0.00 - 1.284		

Analytical Products Group, Inc.

PERFORMANCE REPORT

WP Performance Summary

October 1999

APG Customer Code: 3655

Analytical Management Lab
Suite B
15130 South Keeler
Olathe, KS 66062

Laboratory of
EXCELLENCE

COMMENTS

Oil & Grease WP Level

The results reported by participating laboratories shows a very broad Standard Deviation. The Relative Standard Deviation of the reported results was 30%. Conversely, the USEPA regression equation used to calculate Acceptance Limits predicts a Relative Standard Deviation of 5.5%. This indicates that there is a problem with either the PT samples or with the EPA calculated limits.

The PT sample True Value and homogeneity was verified prior to sample shipment. Based upon the interlaboratory data, the sample was re-tested by APG at the conclusion of the study. The post study data confirmed that the sample was stable and still homogeneous. Both pre-shipment and post shipment analyses were performed by non-EPA methods which have significantly better precision and are designed to confirm the actual materials used in sample preparation. Therefore, APG believes that the PT sample as shipped and tested was stable and correctly prepared.

The USEPA requires PT providers to calculate Acceptance Limits based upon a set of specified regression equations which are provided in the National Standards for Proficiency Testing. These equations predict a linear relationship between the True Value and the Standard Deviation. As noted above, in this case the predicted RSD is 5.5%. However, it is well known that the Relative Standard Deviation of any analysis actually increases near the lower end of the effective concentration range. APG has operated PT programs for more than fifteen years and we have seen similar high percent RSD's on all Oil & Grease PT studies at or below 10 mg/L. This suggests that the method fails below this level.

It should also be noted that although the USEPA published the required regression equations in the National Standards, that they failed to provide any estimate of the quality of these equations.

As a result of the poor correlation between the predicted Standard Deviation and the actual laboratory Standard Deviation almost 50% of the laboratories running this sample failed to meet the EPA limits. The National Standards require that the PT providers prepare samples in the range of 8-50 mg/L and that concentrations vary over the entire range. It should be expected that high failure rates will occur on all samples below 10 mg/L.

Unacceptable	Data more than 3 Deviations from the Mean. Data outside the 99% Confidence Interval, outside the EPA Acceptance Limits.
Blank	No data was reported for this parameter.
Acceptable	Data within acceptable limits.

These notations can be used as a guideline in evaluating your performance.

Comment Section: This section contains comments submitted to Analytical Products Group by program participants. It also contains comments from APG to participants concerning unusual characteristics of the data set or problems with standards which could influence the interpretation of the data.

Performance Evaluation Summary: This section summarizes performance data in a format similar to the USEPA DMR-QA Report. Detailed information on each parameter is included in the main report.

Certification: This program meets the requirements of the ISO/REMCO Harmonized Protocol for Proficiency Testing of Analytical Laboratories as well as the requirements of ASTM E1301. The statistical techniques used to evaluate the data are consistent with ISO and ASTM guidelines and the National Standards for Water Proficiency Testing Programs. APG is an ISO9001 Certified provider of Proficiency Testing Programs.

Introduction to Laboratory Benchmarking System:

The enclosed report includes a ranking summary of your laboratory's performance in the PT program. Several different measures of performance are provided and each should be interpreted separately. The following sections provide a description of the various ranking methods and their interpretation. These rankings are based upon a broad group of laboratories which analyzed the same samples as your laboratory. Laboratories in the program analyzed the samples as single blind PT samples.

Percent Acceptable Data:

The percent acceptable data is calculated as the percent of actual results reported which were within 99% confidence limits for the parameter. Results are reported for your laboratory and the overall average percent acceptable data achieved by all laboratories.

Rank on Percent Acceptable Data:

This is your laboratory's relative rank out of all possible ranks of in the current study. It should be noted that several laboratories can achieve the same rank. The total number of laboratories reporting data for this study is also reported.

Average Percentile: The report includes your laboratory's rank on each parameter and the related percentile rank for that parameter. The average percentile is the average of this data across all parameters reported. It is a different measure of performance than percent acceptable data since it is effected by your relative performance on each parameter. The report provides an average for this measure for all laboratories reporting as well as your laboratory's performance.

Weighted Rank on Percent Acceptable Data:

Because the database includes laboratories of many sizes and capabilities, APG has provided a weighted rank based upon percent acceptable. This measure considers the number of parameters which were run by each laboratory and weights the rank by the total number of parameters evaluated.

Weighted Rank on Average Percentile Data:

This measure also considers the number of parameters run by the laboratory. The weighted rank on percentile data considers not only if the analysis was within the limits but also how close to the Mean the reported result was and the total number of analyses run by the laboratory.

Philip C. Coyner
Proficiency Program Manager

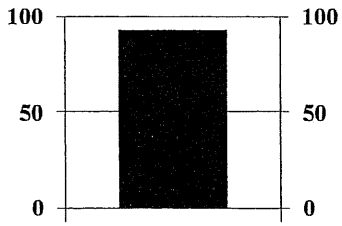
Laboratory Benchmark Report
Summary of Ranking Data

Summary of National Ranking Data

Total Number of Laboratories Reporting:	249
Average Percent Acceptable Data:	92.36%
Average Percentile for all Parameters:	63.58%

National Avg % Acceptable

92.4 %

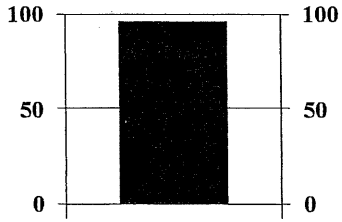


Summary of Laboratory Ranking:

Total Data Points Reported:	125
Number of Acceptable Data Points::	119
Percent Acceptable Data:	95.20%
Average Percentile for all Parameters:	70.13%

Laboratory Avg % Acceptable

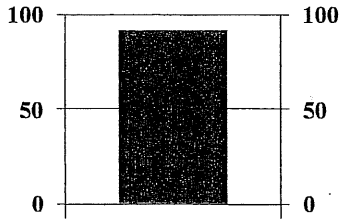
95.20%



Rank on Percent Acceptable Data:	30 of 91
Rank on Average Percentile Data:	98 of 228
Weighted Rank on Percent Acceptable Data:	27 of 125
Weighted Rank on Average Percentile Data:	21 of 241

Weighted Rank Avg Percentile

91.3 %



APG Customer Code 3655
EPA Lab Code KS00902

Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Summary Report

WP October 1999
Study Closing Date 11/15/1999

Product: Acid Extractables

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
2,4,6-Trichlorophenol	WP		34.4	43.4	11.1 - 57.5	0.013	8270C	Acceptable
2,4,6-Trichlorophenol	APG+			136	16.8 - 203			
2,4-Dichlorophenol	WP		20.3	24.2	12.9 - 27.5	0.041	8270C	Acceptable
2,4-Dichlorophenol	APG+			93.1	22.6 - 129			
2,4-Dimethylphenol	WP		20.6	40	8.41 - 53.1	1.37	8270C	Acceptable
2,4-Dimethylphenol	APG+			89.1	20 - 126			
2,4-Dinitrophenol	WP		61.5	72.2	0 - 97.1	0.88	8270C	Acceptable
2,4-Dinitrophenol	APG+			179	24.6 - 236			
2-Chlorophenol	WP		33.1	36.9	17.2 - 43.1	0.67	8270C	Acceptable
2-Chlorophenol	APG+			184	38.3 - 252			
2-Methyl-4,6-Dinitrophenol	WP		80.3	68.8	0 - 107	1.54	8270C	Acceptable
2-Methyl-4,6-Dinitrophenol	APG+			149	43.3 - 193			
2-Nitrophenol	WP		16.6	18.7	5.16 - 23.9	0.68	8270C	Acceptable
2-Nitrophenol	APG+			149	35.8 - 195			
4-Chloro-3-methylphenol	WP		20.8	23.8	14.5 - 26	0.31	8270C	Acceptable
4-Chloro-3-methylphenol	APG+			166	57.4 - 219			
4-Nitrophenol	WP		21.3	95.6	0 - 132	1.55	8270C	Acceptable
4-Nitrophenol	APG+			205	0 - 288			
Pentachlorophenol	WP		54.6	54.4	5.05 - 82.2	0.85	8270C	Acceptable
Pentachlorophenol	APG+			191	0 - 312			
Phenol	WP		10.7	27.2	1 - 34.5	1.25	8270C	Acceptable
Phenol	APG+			104	4 - 129			

APG Customer Code 3655
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Analytical Management Lab
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Olathe, KS 66062

Summary Report

WP October 1999
Study Closing Date 11/15/1999

Product: Base Neutral Extractables

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
1,2,4-Trichlorobenzene	WP		27.4	33.4	5.51 - 45.7	0.27	8270C	Acceptable
1,2,4-Trichlorobenzene	APG+			118	26.1 - 143			
1,2-Dichlorobenzene	WP	94	39.8	48.7	17.5 - 57.5	0.35	8270C	Acceptable
1,2-Dichlorobenzene	APG+	94		159	43.5 - 189			
1,3-Dichlorobenzene	WP		37.9	49.9	7.33 - 64.1	0.23	8270C	Acceptable
1,3-Dichlorobenzene	APG+			210	24.2 - 255			
1,4-Dichlorobenzene	WP		42.6	55.9	11.1 - 69.1	0.26	8270C	Acceptable
1,4-Dichlorobenzene	APG+			161	27.8 - 192			
2,4-Dinitrotoluene (2,4-DNT)	WP		25.2	26.9	9.46 - 35.7	0.59	8270C	Acceptable
2,4-Dinitrotoluene (2,4-DNT)	APG+			64.1	28.3 - 77.8			
2,6-Dinitrotoluene (2,6-DNT)	WP		24.0	28.4	14.8 - 32.8	0.067	8270C	Acceptable
2,6-Dinitrotoluene (2,6-DNT)	APG+			64.9	34.2 - 77.7			
Acenaphthene	WP		21.0	24.3	14.8 - 27.9	0.18	8270C	Acceptable
Acenaphthene	APG+			71.3	39.3 - 83.5			
Anthracene	WP		24.6	27.6	17.6 - 31.3	0.044	8270C	Acceptable
Anthracene	APG+			76.3	40.1 - 92.1			
Bis(2-Ethylhexyl)phthalate	WP		65.6	72.2	17.3 - 100	0.49	8270C	Acceptable
Bis(2-Ethylhexyl)phthalate	APG+			115	35.4 - 161			
Butyl benzyl phthalate	WP		33.1	35.2	0 - 58.8	0.52	8270C	Acceptable
Butyl benzyl phthalate	APG+			218	0 - 319			
Di-n-butyl phthalate	WP		48.5	47.5	13.5 - 66.9	0.93	8270C	Acceptable
Di-n-butyl phthalate	APG+			174	32.1 - 232			

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Olathe, KS 66062

Summary Report

WP October 1999
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Product: Base Neutral Extractables

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Diethyl phthalate	WP		29.2	29.1	1.99 - 44.6	0.83	8270C	Acceptable
Diethyl phthalate	APG+			126	2.7 - 182			
Dimethyl phthalate	WP		48.5	57.3	0 - 92.4	0.56	8270C	Acceptable
Dimethyl phthalate	APG+			133	0 - 214			
Fluoranthene	WP		23.4	23.1	15.5 - 26.2	1.4	8270C	Acceptable
Fluoranthene	APG+			101	53.1 - 123			
Fluorene	WP		28.6	31.9	18.3 - 37.3	0.25	8270C	Acceptable
Fluorene	APG+			117	55.3 - 141			
Hexachlorobenzene	WP		22.9	21.8	6.76 - 31.1	0.99	8270C	Acceptable
Hexachlorobenzene	APG+			60	33.7 - 71.6			
Hexachlorobutadiene	WP		32.1	40.3	4.21 - 52.8	0.44	8270C	Acceptable
Hexachlorobutadiene	APG+			140	26.9 - 163			
Hexachlorocyclopentadiene	WP		29.5	33.3	0 - 51.6	0.69	8270C	Acceptable
Hexachlorocyclopentadiene	APG+			93.5	0 - 128			
Hexachloroethane	WP		19.6	24.6	2.06 - 31.7	0.55	8270C	Acceptable
Hexachloroethane	APG+			60.5	10 - 72.9			
Isophorone	WP		28.0	29	14.6 - 35.8	0.79	8270C	Acceptable
Isophorone	APG+			84.1	27.6 - 110			
Naphthalene	WP		26.3	30.8	10.9 - 38.7	0.33	8270C	Acceptable
Naphthalene	APG+			126	36.3 - 158			
Nitrobenzene (NB)	WP		27.8	42.5	18.8 - 49.2	1.23	8270C	Acceptable
Nitrobenzene (NB)	APG+			93.9	31.7 - 113			

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Olathe, KS 66062

Summary Report

WP October 1999
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Product: Base Neutral Extractables

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Phenanthrene	WP		23.4	24.5	15.9 - 28.2	0.64	8270C	Acceptable
Phenanthrene	APG+			87.9	45.4 - 106			
Pyrene	WP		25.3	27.4	15.3 - 35.9	0.087	8270C	Acceptable
Pyrene	APG+			121	34 - 177			

Product: BTEX

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Benzene	WP		7.97	7.5	5.94 - 9.77	0.17	8260B	Acceptable
Ethylbenzene	WP		15.6	15.7	12 - 18.6	0.27	8260B	Acceptable
m-Xylene / p-Xylene	WP		32.4	31.2	22.6 - 40.2	0.34	8260B	Acceptable
o-Xylene	WP		15.4	15.7	11.6 - 19.8	0.22	8260B	Acceptable
Toluene	WP		8.14	7.88	5.68 - 9.9	0.5	8260B	Acceptable
Total Xylenes	WP		47.8	46.9	31.5 - 61.4	0.28	8260B	Acceptable

Product: Chlordane (Total)

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Chlordane (Total)	WP	53	8.68	8.33	3.64 - 11.9	0.68	8081A	Acceptable

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WP October 1999
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Product: Demand

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Biochemical Oxygen Demand	WP	38	112	106	51 - 151	0.67	405.1	Acceptable
Carboneous BOD	WP	102		88	32.2 - 112			
Chemical Oxygen Demand	WP	36	155	171	132 - 195	0.76	410.1	Acceptable
Total Organic Carbon	WP	37		64.5	53.9 - 74.2			

Product: Minerals

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Alkalinity as CaCO3	WP	27	26.8	23.8	19.8 - 28.8	1.67	310.1	Acceptable
Calcium	WP	23	15.7	16	14 - 18.5	0.81	6010B	Acceptable
Chloride	WP	28	74	65.3	58.6 - 71.5	4.14	300.0	**Not Acceptable**
Conductivity umho/cm	WP	20	264	328	281 - 327	5.25	120.1	**Not Acceptable**
Fluoride	WP	29		0.9	0.76 - 1.03			
Magnesium	WP	24	8.62	9.04	7.85 - 10.2	1.03	6010B	Acceptable
Potassium	WP	26	11.1	11.5	9.75 - 13.3	0.67	6010B	Acceptable
Sodium	WP	25	17.4	16.9	15 - 19.3	0.28	6010B	Acceptable
Sulfate	WP	30	11.1	10.2	7.23 - 12.9	1.06	300.0	Acceptable
Total Dissolved Solids	WP	21	183	181	118 - 212	1.15	160.1	Acceptable
Total Hardness as CaCO3	WP	22	74.7	77.3	68.9 - 86.3	1	6010B	Acceptable

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Olathe, KS 66062

Summary Report

WP October 1999
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Product: Nutrient

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Ammonia Nitrogen as N	WP	31	8.58	8.64	6.68 - 10.5	0.031	350.1	Acceptable
Nitrate Nitrogen as N	WP	32	3.87	4.12	3.23 - 4.93	0.75	300.0	Acceptable
Orthophosphate as P	WP	33	5.09	4.99	4.27 - 5.76	0.32	365.1	Acceptable
Total Kjeldahl Nitrogen	WP	34	4.23	1.88	1.05 - 2.79	7.97	351.2	**Not Acceptable**
Total Phosphorus as P	WP	35	2.22	2.68	2.04 - 3.17	2	365.1	**Check for Error**

Product: Oil & Grease

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Oil & Grease	WP	104	15	9.05	7.04 - 9.77	14.3	1664	**Not Acceptable**

Product: PCB's in Oil

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aroclor 1016/1242 in Oil Sample 2	WP	99	27.0	31.7	5.22 - 43.8	0.39	8082	Acceptable
Aroclor 1254 in Oil Sample 1	WP	100	18.0	17.6	0.39 - 28.6	0.75	8082	Acceptable

Product: PCB's in Water

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aroclor 1016/1242 Sample 2	WP	40	3.58	5.54	1.79 - 7.62	1.15	8082	Acceptable
Aroclor 1254 Sample 1	WP	45	2.88	3.31	1.48 - 4.45	0.18	8082	Acceptable

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WP October 1999
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Product: Pesticides

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
4,4'-DDD	WP	49	1.08	1.06	0.51 - 1.49	0.5	8081A	Acceptable
4,4'-DDE	WP	50	2.43	2.45	1.16 - 3.34	0.5	8081A	Acceptable
4,4'-DDT	WP	51	3.53	3.62	1.65 - 4.9	0.48	8081A	Acceptable
Aldrin	WP	47	0.968	1.2	0.31 - 1.63	0.009	8081A	Acceptable
Dieldrin	WP	48	1.43	1.54	0.79 - 2.13	0.14	8081A	Acceptable
Heptachlor	WP	52	2.45	2.86	0.75 - 3.98	0.15	8081A	Acceptable
Heptachlor epoxide	WP	78	0.73	0.84	0.43 - 1.09	0.27	8081A	Acceptable

Product: pH

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
pH	WP	19	8.345	8.44	8.19 - 8.68	1.16	150.1	Acceptable

Product: Solids

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Dissolved Solids	WP		118	140	106 - 176	1.99	160.1	Acceptable
Total Solids	WP		132	183	155 - 209	5.47	160.3	**Not Acceptable**
Total Suspended Solids	WP	72	39	43	31.2 - 47.2	0.075	160.2	Acceptable

Summary Report

Product: Total Cyanide

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Cyanide	WP	71	0.385	0.47	0.33 - 0.61	1.77	335.3	Acceptable

Product: Total Phenolics

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Phenolics	WP	97	0.46	1	0.36 - 0.97	2	335.3	Check for Error

Product: Total Residual Chlorine

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Residual Chlorine	WP	98		0.76	0.59 - 0.98			

Product: Toxaphene

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Toxaphene	WP		3:22	3.26	0.26 - 6.77	0.27	8081A	Acceptable

Product: TPH Diesel (DRO)

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
TPH Diesel (DRO)	WP		0.293	0.37	0 - 0.55	0.34	8015/OA2	Acceptable

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Product: TPH Gasoline (GRO)

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
TPH Gasoline (GRO)	WP		0.63	0.57	0.11 - 0.76	1.73	8015/0A1	Acceptable

Product: Trace Metals

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aluminum	WP	1	2790	2710	2330 - 3060	0.74	6010B	Acceptable
Antimony	WP	16	145	159	102 - 195	0.26	200.9	Acceptable
Arsenic	WP	2	68.6	75.2	58.8 - 91.1	1.17	200.9	Acceptable
Barium	WP		592	599	541 - 658	0.36	6010B	Acceptable
Beryllium	WP	3	415	428	364 - 483	0.45	6010B	Acceptable
Boron	WP			349	315 - 415			
Cadmium	WP	4	200	204	173 - 232	0.31	6010B	Acceptable
Chromium	WP	6	204	195	168 - 222	1	6010B	Acceptable
Cobalt	WP	5	295	300	263 - 337	0.4	6010B	Acceptable
Copper	WP	7	566	559	507 - 614	0.34	6010B	Acceptable
Iron	WP	8	1080	1080	952 - 1220	0	6010B	Acceptable
Lead	WP	12	1510	1510	1330 - 1680	0	6010B	Acceptable
Manganese	WP	10	102	105	92.8 - 117	0.74	6010B	Acceptable
Mercury	WP	9	3.15	4.82	3.52 - 6.1	3.86	7470	**Not Acceptable**
Molybdenum	WP	74	57.3	60.9	49.7 - 70.8	0.85	6010B	Acceptable
Nickel	WP	11	1910	1810	1640 - 2010	1.29	6010B	Acceptable
Selenium	WP	13	292	291	228 - 338	0.49	6010B	Acceptable

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WP October 1999
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Product: Trace Metals

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Silver	WP	17	318	306	262 - 351	0.75	6010B	Acceptable
Strontium	WP	75		281	239 - 323			
Thallium	WP	18	558	531	428 - 617	1.14	200.9	Acceptable
Titanium	WP	76		144	123 - 163			
Vanadium	WP	14	3000	3010	2720 - 3310	0.1	6010B	Acceptable
Zinc	WP	15	1500	1500	1330 - 1680	0.17	6010B	Acceptable

Product: Volatiles

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
1,1,1-Trichloroethane	WP	56	33.6	32.9	21.4 - 43.4	0.33	8260B	Acceptable
1,1,2,2-Tetrachloroethane	WP		46.1	45.4	31.1 - 60.1	0.1	8260B	Acceptable
1,1,2-Trichloroethane	WP		12.8	12.4	8.99 - 16.5	0.08	8260B	Acceptable
1,1-Dichloroethane	WP		9.23	8.87	6.56 - 11.8	0.058	8260B	Acceptable
1,1-Dichloroethene	WP		23.0	20	12.2 - 32.6	0.18	8260B	Acceptable
1,2-Dichlorobenzene	WP	94	11.1	11.4	7.72 - 15.1	0.24	8260B	Acceptable
1,2-Dichloroethane	WP	54	16.9	15.7	11.1 - 21.5	0.35	8260B	Acceptable
1,2-Dichloropropane	WP		29.3	31.5	22.2 - 39.6	0.55	8260B	Acceptable
1,3-Dichlorobenzene	WP	96	22.5	23.3	16.3 - 29.3	0.14	8260B	Acceptable
1,4-Dichlorobenzene	WP	95	12.4	12.4	8.35 - 16.5	0	8260B	Acceptable
Benzene	WP	65	25.6	24.8	17.7 - 32.3	0.25	8260B	Acceptable
Bromodichloromethane	WP	60	13.2	13.1	9.05 - 17.3	0	8260B	Acceptable

Summary Report

Product: Volatiles

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Bromoform	WP	62	13.8	14.5	8.57 - 20.1	0.26	8260B	Acceptable
Carbon tetrachloride	WP	58	13.3	13.2	8.41 - 18.6	0.12	8260B	Acceptable
Chlorobenzene	WP	64	19.3	20	14.6 - 25.3	0.34	8260B	Acceptable
Chloroform	WP	55	12.2	11.5	8.3 - 15	0.54	8260B	Acceptable
cis-1,3-Dichloropropene	WP		18.3	20.1	13 - 25.5	0.43	8260B	Acceptable
Dibromochloromethane	WP	61	21.3	21.1	12.8 - 28.8	0.19	8260B	Acceptable
Ethylbenzene	WP	66	31.7	32.2	21.6 - 42	0.03	8260B	Acceptable
Methylene chloride	WP	63	23.0	20.2	12.8 - 28.4	0.92	8260B	Acceptable
Tetrachloroethene	WP	59	14.4	14.2	9.31 - 18.6	0.26	8260B	Acceptable
Toluene	WP	67	13.5	14	9.74 - 18	0.29	8260B	Acceptable
trans-1,2-Dichloroethene	WP		9.75	8.41	5.24 - 12.4	0.78	8260B	Acceptable
trans-1,3-Dichloropropene	WP		10.1	10.7	5.75 - 14.9	0.13	8260B	Acceptable
Trichloroethene	WP	57	41.1	40.9	26.6 - 53	0.3	8260B	Acceptable

Exception Report

Product: Minerals

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Chloride	WP	28	74	65.270	58.6 - 71.5	4.14	300.0	**Not Acceptable**
Conductivity umho/cm	WP	20	264	327.800	281 - 327	5.25	120.1	**Not Acceptable**

Product: Nutrient

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Kjeldahl Nitrogen	WP	34	4.23	1.882	1.05 - 2.79	7.97	351.2	**Not Acceptable**

Product: Oil & Grease

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Oil & Grease	WP	104	15	9.054	7.04 - 9.77	14.3	1664	**Not Acceptable**

Product: Solids

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Solids	WP		132	182.900	155 - 209	5.47	160.3	**Not Acceptable**

Product: Trace Metals

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Mercury	WP	9	3.15	4.819	3.52 - 6.1	3.86	7470	**Not Acceptable**

Analytical Products Group, Inc.

PERFORMANCE REPORT

WP Performance Summary

April 2000

APG Customer Code: 3655

Analytical Management Lab
Suite B
15130 South Keeler
Olathe, KS 66062

Laboratory of
EXCELLENCE

2730 Washington Boulevard • Belpre, Ohio 45714 • 800.272.4442 • Fax 740.423.5588

Introduction:
The APG Proficiency Testing Program has been designed to evaluate your laboratory's performance against other participating laboratories on the same set of standards. Sample levels have been chosen to assist you in ensuring that the procedure is in-control. In most cases, sample levels are not designed to challenge the detection limits of the method.

The following information is provided to assist you in the interpretation of the report. All statistical measures have been calculated based upon generally accepted formulae.

Your Result: Every effort has been made to insure correct data entry. Should you find a data entry error please notify Analytical Products Group and we will adjust the data and reissue the report.

Assigned Value: The reported Assigned Value is the calculated True Value of the standard based upon the actual composition of the standard.

Certified Value: Verification of the Assigned Value based upon analysis by APG versus a NIST SRM or an external standard if a NIST SRM is not available. The reported Uncertainty is expressed as $2s/\sqrt{n}$.

EPA Mean: This value is the Mean value calculated as required by the US EPA National Standards for Water Proficiency Testing Program or is the actual Mean for non-EPA parameters

EPA Standard Deviation: This parameter is also calculated based upon the National Standards for Water Proficiency Testing Program or is the actual Standard Deviation for non-EPA parameters

Solids Standards: Solids standards are individually prepared and each standard has a unique Assigned Value. The EPA Mean and Standard Deviation are calculated on the appropriate Assigned Value.

Deviation from the Mean or z score: This result is an expression of how far your reported value was from the Mean of all reporting laboratories in terms of number of Standard Deviations. As a point of reference, EPA Warning Limits are 2 deviations from the Mean and EPA Acceptance Limits are 3 deviations from the Mean.

Evaluation Line: Following the Deviation from the Mean for each parameter is an Evaluation line. The Evaluation line may have one of five entries:

Entry	Description
Unusable	A less than or text value was reported which could not be evaluated.
Check for Error	Data between 2 and 3 Deviations from the Mean. Data outside the 95% Confidence Interval but within EPA Acceptance Limits.

Unacceptable	Data more than 3 Deviations from the Mean. Data outside the 99% Confidence Interval, outside the EPA Acceptance Limits.
Blank	No data was reported for this parameter.
Acceptable	Data within acceptable limits.

These notations can be used as a guideline in evaluating your performance.

Comment Section: This section contains comments submitted to Analytical Products Group by program participants. It also contains comments from APG to participants concerning unusual characteristics of the data set or problems with standards which could influence the interpretation of the data.

Performance Evaluation Summary: This section summarizes performance data in a format similar to the USEPA DMR-QA Report. Detailed information on each parameter is included in the main report.

Certification: This program meets the requirements of the ISO/REMCO Harmonized Protocol for Proficiency Testing of Analytical Laboratories as well as the requirements of ASTM E1301. The statistical techniques used to evaluate the data are consistent with ISO and ASTM guidelines and the National Standards for Water Proficiency Testing Programs. APG is an ISO9001 Certified provider of Proficiency Testing Programs.

Introduction to Laboratory Benchmarking System:

The enclosed report includes a ranking summary of your laboratory's performance in the PT program. Several different measures of performance are provided and each should be interpreted separately. The following sections provide a description of the various ranking methods and their interpretation. These rankings are based upon a broad group of laboratories which analyzed the same samples as your laboratory. Laboratories in the program analyzed the samples as single blind PT samples.

Percent Acceptable Data:

The percent acceptable data is calculated as the percent of actual results reported which were within 99% confidence limits for the parameter. Results are reported for your laboratory and the overall average percent acceptable data achieved by all laboratories.

Rank on Percent Acceptable Data:

This is your laboratory's relative rank out of all possible ranks of in the current study. It should be noted that several laboratories can achieve the same rank. The total number of laboratories reporting data for this study is also reported.

Average Percentile: The report includes your laboratory's rank on each parameter and the related percentile rank for that parameter. The average percentile is the average of this data across all parameters reported. It is a different measure of performance than percent acceptable data since it is effected by your relative performance on each parameter. The report provides an average for this measure for all laboratories reporting as well as your laboratory's performance.

Weighted Rank on Percent Acceptable Data:

Because the database includes laboratories of many sizes and capabilities, APG has provided a weighted rank based upon percent acceptable. This measure considers the number of parameters which were run by each laboratory and weights the rank by the total number of parameters evaluated.

Weighted Rank on Average Percentile Data:

This measure also considers the number of parameters run by the laboratory. The weighted rank on percentile data considers not only if the analysis was within the limits but also how close to the Mean the reported result was and the total number of analyses run by the laboratory.

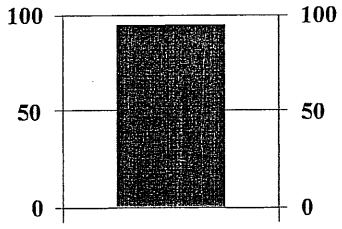
Philip C. Coyner
Proficiency Program Manager

Laboratory Benchmark Report
Summary of Ranking Data

Summary of National Ranking Data

Total Number of Laboratories Reporting:	178
Average Percent Acceptable Data:	94.76%
Average Percentile for all Parameters:	63.02%

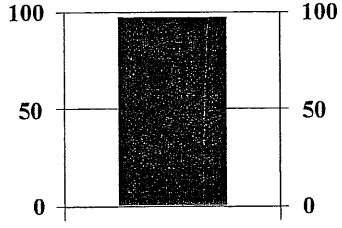
National Avg % Acceptable
94.8 %



Summary of Laboratory Ranking:

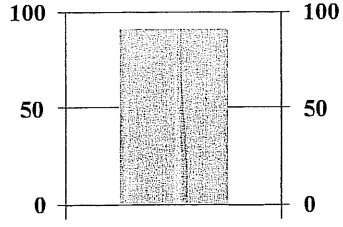
Total Data Points Reported:	130
Number of Acceptable Data Points::	126
Percent Acceptable Data:	96.92%
Average Percentile for all Parameters:	64.58%

Laboratory Avg % Acceptable
96.92%



Rank on Percent Acceptable Data:	22 of 67
Rank on Average Percentile Data:	98 of 167
Weighted Rank on Percent Acceptable	16 of 96
Weighted Rank on Average Percentile Data:	16 of 172

Weighted Rank Avg Percentile
90.7 %



APG Customer 3655
EPA Lab Code KS00902

Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Performance Summary

WP April 2000
Study Closing Date 05/18/2000

Product: Acid Extractables

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
2,4-Dimethylphenol	WP		21.5	24.5	7.21-28.3	1.08	8270C	Acceptable
2-Chlorophenol	WP		38.5	43.8	12.4-53.9	0.77	8270C	Acceptable
2-Methyl-4,6-Dinitrophenol	WP		74.3	75.1	13.1-91	1.71	8270C	Acceptable
2-Nitrophenol	WP		38.3	44.5	9.91-54.9	0.79	8270C	Acceptable
4-Chloro-3-methylphenol	WP		9.70	12.1	4.51-13.3	0.56	8270C	Acceptable
4-Nitrophenol	WP		12.8	52.5	0-65	0.99	8270C	Acceptable
Pentachlorophenol	WP		88.5	92	36-109	1.34	8270C	Acceptable
Phenol	WP		21.0	47.5	0-58	0.46	8270C	Acceptable

Product: Aluminum High Level

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aluminum High Level	WP		0.762	0.75	0.67-0.81	0.96	6010B	Acceptable

APG Customer 3655
EPA Lab Code KS00902

Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Performance Summary

WP April 2000
Study Closing Date 05/18/2000

Product: Base Neutral Extractables

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
1,2,4-Trichlorobenzene	WP		33.5	42.8	9.79-53.2	0.28	8270C	Acceptable
1,2-Dichlorobenzene	WP	94	44.3	57.9	19.7-68.5	0.025	8270C	Acceptable
1,3-Dichlorobenzene	WP		63.8	87.7	10.9-109	0.24	8270C	Acceptable
1,4-Dichlorobenzene	WP		19.4	25	4.28-30.9	0.41	8270C	Acceptable
2,4-Dinitrotoluene (2,4-DNT)	WP		19.6	22.9	8.57-25.6	0.88	8270C	Acceptable
2,6-Dinitrotoluene (2,6-DNT)	WP		30.8	35.7	7.36-47.1	0.54	8270C	Acceptable
Acenaphthene	WP		21.1	22.8	6.62-30.8	0.6	8270C	Acceptable
Anthracene	WP		41.1	40.2	17.8-52.6	1.02	8270C	Acceptable
Benzo(a)anthracene	WP		22.9	25	10.3-32.5	0.4	8270C	Acceptable
Bis(2-Ethylhexyl)phthalate	WP		47.4	50.2	0.72-86.9	0.25	8270C	Acceptable
Butyl benzyl phthalate	WP		66.5	85	15.1-123	0.15	8270C	Acceptable
Di-n-butyl phthalate	WP		40.6	39.8	17.4-51.1	1.14	8270C	Acceptable
Diethyl phthalate	WP		73.8	76.5	17-101	1.08	8270C	Acceptable
Dimethyl phthalate	WP		59.9	91.3	6.32-124	0.27	8270C	Acceptable
Fluoranthene	WP		30.9	30.9	14.3-39.3	0.98	8270C	Acceptable
Fluorene	WP		36.9	41.1	14.7-54.1	0.38	8270C	Acceptable
Hexachlorobenzene	WP		29.3	31.7	13.7-39.4	0.63	8270C	Acceptable
Hexachlorobutadiene	WP		27.1	43.9	8.33-49.7	0.28	8270C	Acceptable
Hexachloroethane	WP		26.6	41.1	3.61-50.9	0.076	8270C	Acceptable
Naphthalene	WP		37.2	44	10.6-56.6	0.47	8270C	Acceptable
Nitrobenzene (NB)	WP		26.1	28.6	6.69-36.9	0.86	8270C	Acceptable
Phenanthrene	WP		19.9	22.8	9.12-30.5	0.028	8270C	Acceptable
Pyrene	WP		31.8	35.5	16.1-47.7	0.019	8270C	Acceptable

APG Customer 3655
EPA Lab Code KS00902

Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Performance Summary

Study Closing Date 05/18/2000

Product: BTEX

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Benzene	WP		18.2	16.1	12-20.8	1.23	8260B	Acceptable
Ethylbenzene	WP		37.8	37.7	27.7-49.1	0.17	8260B	Acceptable
m-Xylene / p-Xylene	WP		47.4	46	33.8-62.8	0.19	8260B	Acceptable
o-Xylene	WP		8.99	8.44	5.69-11.8	0.25	8260B	Acceptable
Toluene	WP		6.62	5.74	4.66-7.29	1.48	8260B	Acceptable
Total Xylenes	WP		56.4	54.5	37.3-74.8	0.048	8260B	Acceptable

Product: Chlordane (Total)

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Chlordane (Total)	WP	53	6.62	7.95	3.47-11.3	0.59	8081A	Acceptable

Product: Demand

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Biochemical Oxygen Demand	WP	38	141	148	71-209	0.038	405.1	Acceptable
Carbonaceous BOD	WP	102		121	44.2-153			
Chemical Oxygen Demand	WP	36	206	227	177-256	0.76	410.1	Acceptable
Total Organic Carbon	WP	37		89.6	74.9-103			

APG Customer 3655
EPA Lab Code KS00902

Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Performance Summary

WP April 2000
Study Closing Date 05/18/2000

Product: Fluoride

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Fluoride	WP	29	0.82	0.9	0.76-1.03	1.56	300.0	Acceptable

Product: Hexavalent Chromium

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Chromium, Hexavalent	WP		18.5	22.3	8.84-34.3	0.73	6010B	Acceptable

Product: Minerals

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Alkalinity as CaCO3	WP	27	68.5	67.6	60.6-74.5	0.39	310.1	Acceptable
Calcium	WP	23	39.0	37.6	33.4-42.7	0.58	6010B	Acceptable
Chloride	WP	28	139	135	123-147	1.01	300.0	Acceptable
Conductivity	WP	20	665	699	606-714	0.28	120.1	Acceptable
Magnesium	WP	24	6.85	6.73	5.82-7.62	0.43	6010B	Acceptable
Potassium	WP	26	32.9	32.8	28.4-37.4	0	6010B	Acceptable
Sodium	WP	25	61.2	59	53.2-64.9	1.13	6010B	Acceptable
Sulfate	WP	30	30.9	32.5	26.1-38	0.6	300.0	Acceptable
Total Dissolved Solids	WP	21	357	366	274-450	0.17	160.1	Acceptable
Total Hardness as CaCO3	WP	22	125.7	122	110-134	0.92	6010B	Acceptable

Performance Summary

WP April 2000
Study Closing Date 05/18/2000

Product: Nutrient

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Ammonia Nitrogen as N	WP	31	12.9	11.5	8.93-14	1.67	350.1	Acceptable
Nitrate Nitrogen as N	WP	32	8.96	8.69	6.86-10.3	0.62	300.0	Acceptable
Orthophosphate as P	WP	33	1.85	2.3	1.96-2.67	3.83	365.1	**Not Acceptable**
Total Kjeldahl Nitrogen	WP	34		8.07	5.68-10.3			
Total Phosphorus as P	WP	35	1.53	1.59	1.2-1.9	0.17	365.1	Acceptable

Product: Oil & Grease

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Oil & Grease	WP	104	25.9	33.3	21.4-38.7	1.42	1664	Acceptable

Product: PCB's in Oil

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aroclor 1254 in Oil Sample 1	WP	100	27.9	37.1	5.59-54.4	0.26	8082	Acceptable
Aroclor 1260 in Oil Sample 2	WP	101	23.4	31.3	5.75-44.3	0.25	8082	Acceptable

Product: PCB's in Water

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aroclor 1016/1242 Sample 1	WP	40	7.28	12.4	3.2-17.7	1.33	8082	Acceptable
Aroclor 1254 Sample 2	WP	45	1.13	1.78	0.68-2.61	1.62	8082	Acceptable

APG Customer 3655
EPA Lab Code KS00902

Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Performance Summary

WP April 2000
Study Closing Date 05/18/2000

Product: Pesticides

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
4,4'-DDD	WP	49	4.36	4.53	2.23-6.31	0.13	8081A	Acceptable
4,4'-DDE	WP	50	3.39	3.72	1.77-5.08	0.055	8081A	Acceptable
4,4'-DDT	WP	51	3.76	3.45	1.57-4.68	1.23	8081A	Acceptable
Aldrin	WP	47	2.25	2.9	0.74-3.91	0.15	8081A	Acceptable
alpha-BHC	WP		4.62	4.95	1.32-7.18	0.38	8081A	Acceptable
beta-BHC	WP		1.99	2.04	0.82-2.86	0.44	8081A	Acceptable
delta-BHC	WP		5.66	4.37	0.87-6.93	1.74	8081A	Acceptable
Dieldrin	WP	48	2.30	2.37	1.22-3.27	0.15	8081A	Acceptable
gamma-BHC	WP		2.55	2.74	0.82-3.96	0.31	8081A	Acceptable
Heptachlor	WP	52	1.59	1.92	0.51-2.67	0	8081A	Acceptable
Heptachlor epoxide	WP	78	1.79	2.18	1.11-2.81	0.61	8081A	Acceptable

Product: pH

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
pH	WP	19	6.576	6.64	6.48-6.81	1.32	150.1	Acceptable

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15130 South Keeler
Olathe, KS 66062

Performance Summary

WP April 2000
Study Closing Date 05/18/2000

Product: Polynuclear Aromatic Hydrocarbons

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Acenaphthene	WP		51.6	46.9	6.74-68.5	1.36	8270C	Acceptable
Anthracene	WP		21.9	21.6	7.91-32.7	0.39	8270C	Acceptable
Fluoranthene	WP		39.7	43	19.5-52.8	0.65	8270C	Acceptable
Fluorene	WP		47.3	44.5	11.1-63.9	1.11	8270C	Acceptable
Naphthalene	WP		20.0	23.4	3.79-30.5	0.63	8270C	Acceptable
Phenanthrene	WP		27.5	33.8	8.9-49.8	0.26	8270C	Acceptable
Pyrene	WP		22.0	24.5	3.9-43.8	0.29	8270C	Acceptable

Product: Solids

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Dissolved Solids	WP		45	43.8	36-53.6	0.068	160.1	Acceptable
Total Solids	WP		79	81.1	68.6-92.6	0.4	160.3	Acceptable
Total Suspended Solids	WP		37	37.3	29.4-40.6	1.08	160.2	Acceptable

Product: Total Cyanide

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Cyanide	WP	71	0.332	0.48	0.33-0.61	2.87	335.3	Check for Error

APG Customer 3655
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Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Performance Summary

WP April 2000
Study Closing Date 05/18/2000

Product: Total Phenolics

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Phenolics	WP	97	0.437	0.91	0.33-0.87	1.79	420.2	Acceptable

Product: Total Residual Chlorine

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Residual Chlorine	WP	98	1.20	0.89	0.69-1.11	4.23	330.4	**Not Acceptable**

Product: Toxaphene

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Toxaphene	WP		4.09	4.3	2.47-6.31	0.47	8081A	Acceptable

Product: TPH 418.1

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
TPH by 418.1	WP			46.3	25.6-97.5			

Product: TPH Diesel (DRO)

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
TPH Diesel (DRO)	WP		2.05	2.6	0.34-3.37	0.37	8015/OA2	Acceptable

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Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Performance Summary

WP April 2000
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Product: TPH Gasoline (GRO)

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
TPH Gasoline (GRO)	WP		2.02	3.21	0-4.99	0.18	8015/0A1	Acceptable

APG Customer 3655
EPA Lab Code KS00902

Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Performance Summary

WP April 2000
Study Closing Date 05/18/2000

Product: Trace Metals

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aluminum	WP	1	2150	2080	1790-2360	0.84	6010B	Acceptable
Antimony	WP	16	305	308	210-373	0.52	200.9	Acceptable
Arsenic	WP	2	199	200	165-236	0.083	200.9	Acceptable
Barium	WP		854	874	770-976	0.55	6010B	Acceptable
Beryllium	WP	3	153	166	140-188	1.39	6010B	Acceptable
Boron	WP			349	313-396			
Cadmium	WP	4	64.3	59.2	49.7-68.5	1.66	6010B	Acceptable
Chromium	WP	6	795	742	647-839	1.62	6010B	Acceptable
Cobalt	WP	5	515	500	439-561	0.74	6010B	Acceptable
Copper	WP	7	105	108	95.2-122	0.9	6010B	Acceptable
Iron	WP	8	107	167	143-194	7.28	6010B	**Not Acceptable**
Lead	WP	12	686	708	619-794	0.72	200.9	Acceptable
Manganese	WP	10	1910	1880	1690-2080	0.3	6010B	Acceptable
Mercury	WP	9	12.1	16.2	12.2-20.2	3.06	7470	**Not Acceptable**
Molybdenum	WP	74	295	277	236-317	1.33	6010B	Acceptable
Nickel	WP	11	451	420	376-472	1.69	6010B	Acceptable
Selenium	WP	13	482	501	396-581	0.23	200.9	Acceptable
Silver	WP	17	285	265	227-304	1.48	6010B	Acceptable
Strontium	WP	75		185	157-212			
Thallium	WP	18	400	403	324-469	0.17	200.9	Acceptable
Titanium	WP	76		201	173-227			
Vanadium	WP	14	3350	3340	3010-3660	0.092	6010B	Acceptable
Zinc	WP	15	347	333	292-378	0.84	6010B	Acceptable

APG Customer 3655
EPA Lab Code KS00902

Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Performance Summary

Study Closing Date 05/18/2000

Product: Volatiles

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
1,1,1-Trichloroethane	WP	56	12.5	11.6	7.86-15.3	0.73	8260B	Acceptable
1,1,2,2-Tetrachloroethane	WP		7.13	7.44	4.65-10.1	0.25	8260B	Acceptable
1,1,2-Trichloroethane	WP		40.7	37.3	33-43.5	1.38	8260B	Acceptable
1,2-Dichlorobenzene	WP	94	10.1	9.59	6.4-12.8	0.48	8260B	Acceptable
1,2-Dichloroethane	WP	54	11.5	10.8	7.65-15	0.16	8260B	Acceptable
1,2-Dichloropropane	WP		9.12	8.58	6-11.2	0.59	8260B	Acceptable
1,3-Dichlorobenzene	WP	96	31.9	31.1	21.9-39	0.52	8260B	Acceptable
1,4-Dichlorobenzene	WP	95	49.7	41.9	28.8-54.1	1.97	8260B	Acceptable
Benzene	WP	65	44.6	41.3	29.7-53.3	0.79	8260B	Acceptable
Bromodichloromethane	WP	60	48.6	46.8	33.2-60.9	0.35	8260B	Acceptable
Bromoform	WP	62	11.4	11.8	6.74-16.4	0.12	8260B	Acceptable
Carbon tetrachloride	WP	58	33.8	33	20.5-46.6	0.046	8260B	Acceptable
Chlorobenzene	WP	64	34.5	32.6	23.3-41.4	0.73	8260B	Acceptable
Chloroform	WP	55	13.0	11.9	8.6-15.6	0.78	8260B	Acceptable
Dibromochloromethane	WP	61	33.9	33.8	21.5-45.2	0.15	8260B	Acceptable
Ethylbenzene	WP	66	11.9	11.1	7.72-14.3	0.82	8260B	Acceptable
Methylene chloride	WP	63	10.9	9.7	6.17-14.2	0.53	8260B	Acceptable
Tetrachloroethene	WP	59	10.7	10.1	6.59-13.4	0.63	8260B	Acceptable
Toluene	WP	67	28.9	26.3	18.7-33.2	1.24	8260B	Acceptable
Trichloroethene	WP	57	24.9	22.9	15-29.8	1.01	8260B	Acceptable

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Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Performance Summary

WP April 2000
Study Closing Date 05/18/2000

Product: Nutrient **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Orthophosphate as P	WP	33	1.85	2.3	1.96-2.67	3.83	365.1	Not Acceptable

Product: Total Residual Chlorine **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Residual Chlorine	WP	98	1.20	0.89	0.69-1.11	4.23	330.4	Not Acceptable

Product: Trace Metals **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Iron	WP	8	107	167	143-194	7.28	6010B	Not Acceptable
Mercury	WP	9	12.1	16.2	12.2-20.2	3.06	7470	Not Acceptable

Analytical Products Group, Inc.

PERFORMANCE REPORT

WP Performance Summary

October 2000

APG Customer Code: 3655

Analytical Management Lab
Suite B
15130 South Keeler
Olathe, KS 66062

Laboratory of
EXCELLENCE

WP Performance Summary Introduction

Introduction:

The APG Proficiency Testing Program has been designed to evaluate your laboratory's performance against other participating laboratories on the same set of standards. Sample levels have been chosen to meet the requirements as specified by the National Standards for Water Proficiency Testing Studies Criteria Document (Document # NERL-Ci-0045) and the NELAP Standards, to assist you in ensuring that the procedure is in-control. In most cases, sample levels are not designed to challenge the detection limits of the method.

The following information is provided to assist you in the interpretation of the report. All statistical measures have been calculated based upon generally accepted formulae.

Your Result:

Every effort has been made to insure correct data entry. Should you find a data entry error, please notify Analytical Products Group and we will adjust the data and reissue the report.

Assigned Value:

The reported Assigned Value is the calculated True Value of the standard based upon the actual composition of the standard.

Certified Value:

Verification of the Assigned Value based upon analysis by APG versus a NIST SRM or an external standard if a NIST SRM is not available. The reported Uncertainty is expressed as 2s/Ön.

EPA Mean:

This value is the Mean value calculated as required by the US EPA National Standards for Water Proficiency Testing Program or is the actual Mean for non-EPA parameters

EPA Standard Deviation:

This parameter is also calculated based upon the National Standards for Water Proficiency Testing Program or is the actual Standard Deviation for non-EPA parameters.

Solids Standards

Solids standards are individually prepared and each standard has a unique Assigned Value. The EPA Mean and Standard Deviation are calculated on

Deviation from the Mean or Z score

This result is an expression of how far your reported value was from the Mean of all reporting laboratories in terms of number of Standard Deviations. As a point of reference, EPA Warning Limits are 2 deviations from the Mean and EPA Acceptance Limits are 3 deviations from the Mean.

Evaluation Line:

Following the Deviation from the Mean for each parameter is an Evaluation line. The Evaluation line may have one of five entries:

'<' Evaluation

We have implemented the '<' reporting and evaluation method as specified in the National Standards for Water Proficiency Testing Criteria Document. This program allows laboratories to have non-detects evaluated as acceptable results if they are properly reported. If your laboratory correctly reports a negative result it will be evaluated as 'Acceptable' if you report a zero (0), or less than (<) value for the parameter. The US EPA Database cannot evaluate any other reported result. Therefore, if you report any other value such as ND, Not Detected, BDL, etc the data will be evaluated as 'Unusable' and you will not receive credit for the result. However, if you report a blank result for a parameter it will not be evaluated in your report. A less than (<) value will only be judged 'Acceptable' for a parameter which is present if the acceptance limit includes zero and the entire range represented by the less than result is included in the acceptance limit.

WP Performance Summary Introduction

Evaluation Line (Cont):

Entry	Description
Unusable	A text value was reported which could not be evaluated
Check for Error	Data between 2 and 3 Deviations from the Mean. Data outside the 95% Confidence Interval but within EPA Acceptance Limits.
Not Acceptable	Data more than 3 Deviations from the Mean. Data outside the 99% Confidence Interval, outside the EPA Acceptance Limits.
Blank	No data was reported for this parameter.
Acceptable	Data within acceptable limits.

These notations can be used as a guideline in evaluating your performance.

Comment Section

This section contains comments submitted to Analytical Products Group by program participants. It also contains comments from APG to participants concerning unusual characteristics of the data set or problems with standards which could influence the interpretation of the data.

Performance Evaluation Summary

This section summarizes performance data in a format similar to the USEPA DMR-QA Report. Detailed information on each parameter is included in the main report.

Certification

This program meets the requirements of the ISO/REMCO Harmonized Protocol for Proficiency Testing of Analytical Laboratories as well as the requirements of ASTM E1301. The statistical techniques used to evaluate the data are consistent with ISO and ASTM guidelines and the National Standards for Water Proficiency Testing Programs. APG is an ISO9001 Certified provider of Proficiency Testing Programs.

Introduction to Laboratory Benchmarking System:

The enclosed report includes a ranking summary of your laboratory's performance in the PT program. Several different measures of performance are provided and each should be interpreted separately. The following sections provide a description of the various ranking methods and their interpretation. These rankings are based upon a broad group of laboratories which analyzed the same samples as your laboratory. Laboratories in the program analyzed the samples as single blind PT samples.

Percent Acceptable Data:

The percent acceptable data is calculated as the percent of actual results reported which were within 99% confidence limits for the parameter. Results are reported for your laboratory and the overall average percent acceptable data achieved by all laboratories.

Rank on Percent Acceptable Data:

This is your laboratory's relative rank out of all possible ranks of in the current study. It should be noted that several laboratories can achieve the same rank. The total number of laboratories reporting data for this study is also reported.

WP Performance Summary Introduction

Average Percentile

The report includes your laboratory's rank on each parameter and the related percentile rank for that parameter. The average percentile is the average of this data across all parameters reported. It is a different measure of performance than percent acceptable data since it is effected by your relative performance on each parameter. The report provides an average for this measure for all laboratories reporting as well as your laboratory's performance.

Weighted Rank on Percent Acceptable

Because the database includes laboratories of many sizes and capabilities, APG has provided a weighted rank based upon percent acceptable. This measure considers the number of parameters which were run by each laboratory and weights the rank by the total number of parameters evaluated.

Weighted Rank on Average Percentile

This measure also considers the number of parameters run by the laboratory. The weighted rank on percentile data considers not only if the analysis was within the limits but also how close to the Mean the reported result was and the total number of analyses run by the laboratory.

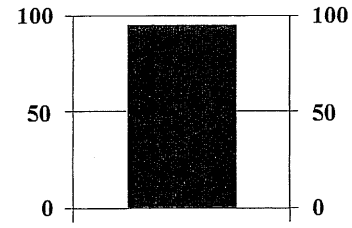
Philip C. Coyner
Proficiency Program Manager

Laboratory Benchmark Report
Summary of Ranking Data

Summary of National Ranking Data

Total Number of Laboratories Reporting:	190
Average Percent Acceptable Data:	94.87%
Average Percentile for all Parameters:	62.42%

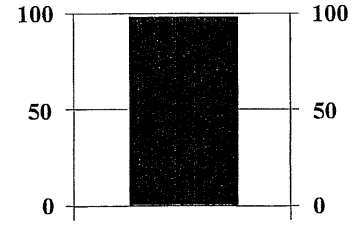
National Avg % Acceptable
94.9 %



Summary of Laboratory Ranking:

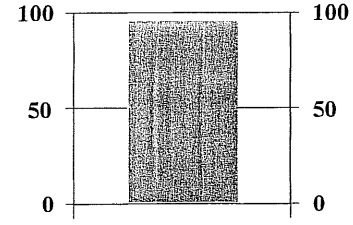
Total Data Points Reported:	189
Number of Acceptable Data Points::	185
Percent Acceptable Data:	97.88%
Average Percentile for all Parameters:	69.40%

Laboratory Avg % Acceptable
97.88%



Rank on Percent Acceptable Data:	18 of 74
Rank on Average Percentile Data:	55 of 171
Weighted Rank on Percent Acceptable	12 of 109
Weighted Rank on Average Percentile Data:	9 of 182

Weighted Rank Avg Percentile
95.1 %



Performance Summary

Product: Acid Extractables **Lot Number: 28865**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
2,4,6-Trichlorophenol	WP		24.1	27.3	9.43-34.8	0.47	8270C	Acceptable
2,4-Dichlorophenol	WP		<10				8270C	Acceptable
2,4-Dimethylphenol	WP		22.3	25	2.64-37	0.44	8270C	Acceptable
2,4-Dinitrophenol	WP		<10				8270C	Acceptable
2-Chlorophenol	WP		29.8	35.9	9.96-45.9	0.32	8270C	Acceptable
2-Methyl-4,6-Dinitrophenol	WP		67.2	78.2	12.2-98.8	0.81	8270C	Acceptable
2-Nitrophenol	WP		<10				8270C	Acceptable
4-Chloro-3-methylphenol	WP		18.4	19.9	6.3-26.3	0.63	8270C	Acceptable
4-Nitrophenol	WP		<10				8270C	Acceptable
Pentachlorophenol	WP		63.0	64.7	23.9-85.1	0.83	8270C	Acceptable
Phenol	WP		15.9	21.6	2.83-25.5	0.45	8270C	Acceptable

Product: Aluminum High Level **Lot Number: 28874**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aluminum High Level	WP		0.46	0.45	0.34-0.55	0.28	6010B	Acceptable

Performance Summary

Product: Base Neutral Extractables **Lot Number: 28866**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
1,2,4-Trichlorobenzene	WP		22.7	30.4	9.02-38.4	0.2	8270C	Acceptable
1,2-Dichlorobenzene	WP	94	31.7	42.1	16-49.7	0.2	8270C	Acceptable
1,3-Dichlorobenzene	WP		68.8	90.8	14.5-112	0.34	8270C	Acceptable
1,4-Dichlorobenzene	WP		39.4	54.1	11.5-66.6	0.033	8270C	Acceptable
2,4-Dinitrotoluene (2,4-DNT)	WP		24.4	28.7	5.65-36.4	0.66	8270C	Acceptable
2,6-Dinitrotoluene (2,6-DNT)	WP		<10				8270C	Acceptable
2-Chloronaphthalene	WP		<10				8270C	Acceptable
3,3'-Dichlorobenzidine	WP		<10				8270C	Acceptable
4-Bromophenyl phenyl ether	WP		<10				8270C	Acceptable
4-Chlorophenyl phenyl ether	WP		<10				8270C	Acceptable
Acenaphthene	WP		18.2	23.1	11.7-28.6	0.71	8270C	Acceptable
Acenaphthylene	WP		<10				8270C	Acceptable
Anthracene	WP		29.9	32.4	18-39.6	0.31	8270C	Acceptable
Benzidine	WP		<10				8270C	Acceptable
Benzo(a)anthracene	WP		<10				8270C	Acceptable
Benzo(a)pyrene	WP		<10				8270C	Acceptable
Benzo(b)fluoranthene	WP		<10				8270C	Acceptable
Benzo(ghi)perylene	WP		<10				8270C	Acceptable
Benzo(k)fluoranthene	WP		<10				8270C	Acceptable
Bis(2-Chloroethoxy)methane	WP		<10				8270C	Acceptable
Bis(2-Chloroethyl)ether	WP		<10				8270C	Acceptable
Bis(2-Chloroisopropyl)ether	WP		<10				8270C	Acceptable
Bis(2-Ethylhexyl)phthalate	WP		23.9	25.6	10.2-36	0.19	8270C	Acceptable

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Butyl benzyl phthalate	WP	63.9	64.7	0-104	0.91	8270C	Acceptable
Chrysene	WP	<10				8270C	Acceptable
Di-n-butyl phthalate	WP	46.1	42.7	15.8-57.5	1.35	8270C	Acceptable
Di-n-octyl phthalate	WP	<10				8270C	Acceptable
Dibenzo(a,h)anthracene	WP	<10				8270C	Acceptable
Diethyl phthalate	WP	85.2	80.9	0-123	1.26	8270C	Acceptable
Dimethyl phthalate	WP	35.5	38.1	0-61.8	0.84	8270C	Acceptable
Fluoranthene	WP	<10				8270C	Acceptable
Fluorene	WP	19.1	23.6	12.1-28.8	0.47	8270C	Acceptable
Hexachlorobenzene	WP	20.0	22.4	14.1-26.3	0.099	8270C	Acceptable
Hexachlorobutadiene	WP	31.9	42.1	8.93-51.9	0.21	8270C	Acceptable
Hexachlorocyclopentadiene	WP	<10				8270C	Acceptable
Hexachloroethane	WP	24.7	35.4	5.61-43.8	0	8270C	Acceptable
Indeno(1,2,3-cd)pyrene	WP	<10				8270C	Acceptable
Isophorone	WP	46.2	41.3	17.9-53.5	1.77	8270C	Acceptable
N-nitrosodi-n-propylamine	WP	<10				8270C	Acceptable
N-nitrosodimethylamine	WP	<10				8270C	Acceptable
N-nitrosodiphenylamine	WP	<10				8270C	Acceptable
Naphthalene	WP	20.4	26.5	11.1-31.9	0.32	8270C	Acceptable
Nitrobenzene (NB)	WP	18.8	21.9	7.4-30.2	0	8270C	Acceptable
Phenanthrene	WP	27.1	31.7	14.9-38.5	0.1	8270C	Acceptable
Pyrene	WP	<10				8270C	Acceptable

Performance Summary

Product: BTEX **Lot Number: 28868**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Benzene	WP		8.39	8.22	5.25-12.1	0.26	8260B	Acceptable
Ethylbenzene	WP		24.7	22.9	16.2-29.8	0.75	8260B	Acceptable
m-Xylene / p-Xylene	WP		<10				8260B	Acceptable
o-Xylene	WP		30.7	28.3	19.7-36.3	0.98	8260B	Acceptable
Toluene	WP		16.4	15.9	11.7-21	0.065	8260B	Acceptable
Total Xylenes	WP		30.7	28.3	15.8-44.7	0.083	8260B	Acceptable

Product: Chlordane (Total) **Lot Number: 28862**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Chlordane (Total)	WP	53	11.4	12	5.25-17.1	0.1	8081A	Acceptable

Product: Demand **Lot Number: 28845**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Biochemical Oxygen Demand	WP	38	64	63.2	31.8-94.6	0.079	405.1	Acceptable
Carbonaceous BOD	WP	102		54.4	24.4-84.4			
Chemical Oxygen Demand	WP	36	77	102	75.8-119	2.84	410.1	Check for Error
Total Organic Carbon	WP	37		40.3	33.6-46.6			

Performance Summary

Product: Fluoride **Lot Number: 28875**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Fluoride	WP	29	0.49	0.21	0.15-0.27	14	300.0	**Not Acceptable**

Product: Hexavalent Chromium **Lot Number: 28864**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Chromium, Hexavalent	WP		29	41.8	24.9-59.5	2.29	7196A	Check for Error

Product: Minerals **Lot Number: 28848-28849**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Alkalinity as CaCO3	WP	27	17.9	17.4	13.9-22.2	0.073	310.1	Acceptable
Calcium	WP	23	12.3	12	10.4-13.9	0.17	6010B	Acceptable
Chloride	WP	28	72.5	73.2	66-80.2	0.25	325.3	Acceptable
Conductivity	WP	20	395	411	357-417	0.8	120.1	Acceptable
Magnesium	WP	24	4.82	4.86	4.18-5.53	0.18	6010B	Acceptable
Potassium	WP	26	14.5	14.9	12.7-17.1	0.54	6010B	Acceptable
Sodium	WP	25	44.2	43.3	38.9-47.8	0.54	6010B	Acceptable
Sulfate	WP	30	47.6	40.8	33.1-47.4	3.11	300.0	**Not Acceptable**
Total Dissolved Solids	WP	21	190	207	146-256	0.6	160.1	Acceptable
Total Hardness as CaCO3	WP	22	50.6	50.1	43.7-57	0.14	6010B	Acceptable

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Product: Nutrient **Lot Number: 28846-28847**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Ammonia Nitrogen as N	WP	31	6.41	6.54	5.03-8	0.2	350.1	Acceptable
Nitrate Nitrogen as N	WP	32	8.63	7.64	6.03-9.1	2.1	300.0	**Check for Error**
Orthophosphate as P	WP	33	4.56	4.54	3.88-5.25	0.044	365.1	Acceptable
Total Kjeldahl Nitrogen	WP	34		10.2	7.31-12.9			
Total Phosphorus as P	WP	35	4.76	4.01	3.05-4.71	3.14	365.3	**Not Acceptable**

Product: Oil & Grease **Lot Number: 28856**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Oil & Grease	WP	104	16.4	17.7	9.86-22.5	0.095	1664	Acceptable

Product: PCB's in Oil **Lot Number: 28859-28860**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aroclor 1016/1242 in Oil Sample 1	WP	99	42.7	37.4	6.65-50.6	1.92	8082	Acceptable
Aroclor 1016/1242 in Oil Sample 2	WP	99	<20				8082	Acceptable
Aroclor 1254 in Oil Sample 1	WP	100	<20				8082	Acceptable
Aroclor 1254 in Oil Sample 2	WP	100	38.1	31.8	4.17-47.4	1.71	8082	Acceptable
Aroclor 1260 in Oil Sample 1	WP	101	<20				8082	Acceptable
Aroclor 1260 in Oil Sample 2	WP	101	<20				8082	Acceptable

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Product: PCB's in Water **Lot Number: 28857-28858**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aroclor 1016/1242 Sample 1	WP	40	5.02	6.08	1.9-8.42	0.13	8082	Acceptable
Aroclor 1016/1242 Sample 2	WP	40	<1				8082	Acceptable
Aroclor 1232 Sample 1	WP	42	<1				8082	Acceptable
Aroclor 1232 Sample 2	WP	42	<1				8082	Acceptable
Aroclor 1248 Sample 1	WP	44	<1				8082	Acceptable
Aroclor 1248 Sample 2	WP	44	1.47	1.62	0.65-2.19	0.19	8082	Acceptable
Aroclor 1254 Sample 1	WP	45	<1				8082	Acceptable
Aroclor 1254 Sample 2	WP	45	<1				8082	Acceptable
Aroclor 1260 Sample 1	WP	46	<1				8082	Acceptable
Aroclor 1260 Sample 2	WP	46	<1				8082	Acceptable

Performance Summary

Product: Pesticides **Lot Number: 28861**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
4,4'-DDD	WP	49	2.56	3.08	1.51-4.3	0.72	8081A	Acceptable
4,4'-DDE	WP	50	0.717	0.82	0.38-1.11	0.28	8081A	Acceptable
4,4'-DDT	WP	51	3.33	4.04	1.84-5.46	0.53	8081A	Acceptable
Aldrin	WP	47	0.566	0.65	0.16-0.87	0.38	8081A	Acceptable
alpha-BHC	WP		<0.05				8081A	Acceptable
beta-BHC	WP		<0.05				8081A	Acceptable
delta-BHC	WP		<0.05				8081A	Acceptable
Dieldrin	WP	48	0.893	1.02	0.53-1.42	0.51	8081A	Acceptable
Endosulfan I	WP		<0.05				8081A	Acceptable
Endosulfan II	WP		<0.05				8081A	Acceptable
Endosulfan sulfate	WP		4.21	4.96	2.76-6.7	0.79	8081A	Acceptable
Endrin	WP		<0.05				8081A	Acceptable
Endrin aldehyde	WP		<0.05				8081A	Acceptable
gamma-BHC	WP		<0.05				8081A	Acceptable
Heptachlor	WP	52	1.35	1.62	0.43-2.25	0.033	8081A	Acceptable
Heptachlor epoxide	WP	78	2.139	2.42	1.23-3.12	0.1	8081A	Acceptable
Methoxychlor	WP		<0.05				8081A	Acceptable

Product: pH **Lot Number: 28850**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
pH	WP	19	7.043	7.11	6.92-7.3	1.06	150.1	Acceptable

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Product: Polynuclear Aromatic Hydrocarbons Lot Number: 28867

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Acenaphthene	WP		21.1	26.7	7.51-34.8	0	8270C	Acceptable
Acenaphthylene	WP		<10				8270C	Acceptable
Anthracene	WP		28.9	33.5	12-48.2	0.2	8270C	Acceptable
Benzo(a)anthracene	WP		<10				8270C	Acceptable
Benzo(a)pyrene	WP		<10				8270C	Acceptable
Benzo(b)fluoranthene	WP		<10				8270C	Acceptable
Benzo(ghi)perylene	WP		<10				8270C	Acceptable
Benzo(k)fluoranthene	WP		<10				8270C	Acceptable
Chrysene	WP		<10				8270C	Acceptable
Dibenzo(a,h)anthracene	WP		<10				8270C	Acceptable
Fluoranthene	WP		27.5	30.6	18.4-35.1	0.29	8270C	Acceptable
Fluorene	WP		32.8	41.5	17.2-52.6	0.36	8270C	Acceptable
Indeno(1,2,3-cd)pyrene	WP		<10				8270C	Acceptable
Naphthalene	WP		16.8	22	7.47-29.3	0.44	8270C	Acceptable
Phenanthrene	WP		23.2	28.5	12.2-38	0.44	8270C	Acceptable
Pyrene	WP		42.2	44.4	17.9-60.5	0.42	8270C	Acceptable

Product: Solids Lot Number: 63760

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Dissolved Solids	WP		57	44.6	28.9-68.9	1.21	160.1	Acceptable
Total Solids	WP		112	107	80.3-138	0.31	160.3	Acceptable
Total Suspended Solids	WP	72	55	62.8	48-67.6	0.86	160.2	Acceptable

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Performance Summary

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Product: Total Cyanide **Lot Number: 28854**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Cyanide	WP	71	0.254	0.57	0.39-0.73	5.37	335.3	**Not Acceptable**

Product: Total Phenolics **Lot Number: 28853**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Phenolics	WP	97	0.224	0.29	0.096-0.28	1.1	420.2	Acceptable

Product: Total Residual Chlorine **Lot Number: 28855**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Residual Chlorine	WP	98	3.28	3.3	2.57-3.71	0.74	330.4	Acceptable

Product: Toxaphene **Lot Number: 28871**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Toxaphene	WP		4.60	3.03	0.82-5.26	2.11	8081A	Check for Error

Product: TPH 418.1 **Lot Number: 28876**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
TPH by 418.1	WP			63	0-172			

Performance Summary

Product: TPH Diesel (DRO) **Lot Number: 28869**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
TPH Diesel (DRO)	WP		4.2	4.83	0-7.23	0.79	8015/OA2	Acceptable

Product: TPH Gasoline (GRO) **Lot Number: 28870**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
TPH Gasoline (GRO)	WP		0.60	0.77	0.035-1.4	0.52	8015/OA1	Acceptable

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Product: Trace Metals **Lot Number: 28851-28852**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aluminum	WP	1	252	250	201-302	0.06	6010B	Acceptable
Antimony	WP	16	458	461	321-556	0.49	6010B	Acceptable
Arsenic	WP	2	154	153	125-182	0.11	200.9	Acceptable
Arsenic	WP	2	158	153	125-182	0.53	6010B	Acceptable
Barium	WP		1765	1750	1570-1930	0.25	6010B	Acceptable
Beryllium	WP	3	263	291	247-329	1.82	6010B	Acceptable
Boron	WP			291	237-373			
Cadmium	WP	4	326	316	269-359	0.8	6010B	Acceptable
Chromium	WP	6	487	464	404-525	1.13	6010B	Acceptable
Cobalt	WP	5	291	280	245-314	0.95	6010B	Acceptable
Copper	WP	7	223	217	194-240	0.78	6010B	Acceptable
Iron	WP	8	1358	1330	1180-1500	0.33	6010B	Acceptable
Lead	WP	12	1839	1770	1560-1970	1	6010B	Acceptable
Manganese	WP	10	769	750	673-833	0.6	6010B	Acceptable
Mercury	WP	9	6.41	5.44	4-6.87	2.04	7470	Check for Error
Molybdenum	WP	74	371	369	316-422	0.11	6010B	Acceptable
Nickel	WP	11	343	319	284-360	1.65	6010B	Acceptable
Selenium	WP	13	816	835	663-967	0.02	6010B	Acceptable
Silver	WP	17	373	349	300-400	1.37	6010B	Acceptable
Strontium	WP	75		99.4	84.2-114			
Thallium	WP	18	224	215	171-252	0.97	200.9	Acceptable
Thallium	WP	18	215	215	171-252	0.3	6010B	Acceptable
Titanium	WP	76		161	138-182			

APG Customer 3655 Analytical Management Lab
EPA Lab Code KS00902 15130 South Keeler
Olathe, KS 66062

Performance Summary

WP October 2000
Study Closing Date 11/16/2000

Vanadium		WP	14	834	834	751-915	0.037	6010B	Acceptable
Zinc		WP	15	309	300	263-341	0.54	6010B	Acceptable

Performance Summary

Product: Volatiles **Lot Number: 28863**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
1,1,1-Trichloroethane	WP	56	25.9	27	17.6-35.6	0.23	8260B	Acceptable
1,1,2,2-Tetrachloroethane	WP		15.7	13.7	8.28-19.9	0.83	8260B	Acceptable
1,1,2-Trichloroethane	WP		8.81	8.42	6.16-10.4	0.73	8260B	Acceptable
1,1-Dichloroethane	WP		<2				8260B	Acceptable
1,1-Dichloroethene	WP		<2				8260B	Acceptable
1,2-Dichlorobenzene	WP	94	21.1	19.4	13.4-25	0.98	8260B	Acceptable
1,2-Dichloroethane	WP	54	39.2	40.1	27.7-53.5	0.33	8260B	Acceptable
1,2-Dichloropropane	WP		70.8	64.7	47-82.1	1.08	8260B	Acceptable
1,3-Dichlorobenzene	WP	96	32.6	30.5	21.4-38.3	1	8260B	Acceptable
1,4-Dichlorobenzene	WP	95	8.05	7.55	5-10.3	0.44	8260B	Acceptable
Benzene	WP	65	15.9	16	11.2-21	0.12	8260B	Acceptable
Bromodichloromethane	WP	60	27.5	25.3	17.8-33.1	0.78	8260B	Acceptable
Bromoform	WP	62	12.3	11.5	6.56-16	0.63	8260B	Acceptable
Carbon tetrachloride	WP	58	10.8	11	7.13-15.7	0.42	8260B	Acceptable
Chlorobenzene	WP	64	13.6	13.3	9.96-16.7	0.27	8260B	Acceptable
Chloroform	WP	55	26.3	26.8	18.8-34.6	0.15	8260B	Acceptable
cis-1,3-Dichloropropene	WP		<2				8260B	Acceptable
Dibromochloromethane	WP	61	40.5	34.8	22.2-46.5	1.51	8260B	Acceptable
Ethylbenzene	WP	66	14.2	14.3	9.81-18.5	0.069	8260B	Acceptable
Methylene chloride	WP	63	43.3	44.2	27.9-61	0.22	8260B	Acceptable
Tetrachloroethene	WP	59	11.5	11.9	7.81-15.7	0.23	8260B	Acceptable
Toluene	WP	67	22.2	22.3	15.8-28.2	0.096	8260B	Acceptable
trans-1,2-Dichloroethene	WP		<2				8260B	Acceptable

APG Customer 3655 Analytical Management Lab
EPA Lab Code KS00902 15130 South Keeler
Olathe, KS 66062

Performance Summary

WP October 2000
Study Closing Date 11/16/2000

trans-1,3-Dichloropropene	WP		<2				8260B	Acceptable
Trichloroethene	WP	57	9.03	9.37	6.24-12.3	0.26	8260B	Acceptable

Performance Summary

Product: Fluoride **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Fluoride	WP	29	0.49	0.21	0.15-0.27	14	300.0	Not Acceptable

Product: Minerals **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Sulfate	WP	30	47.6	40.8	33.1-47.4	3.11	300.0	Not Acceptable

Product: Nutrient **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Phosphorus as P	WP	35	4.76	4.01	3.05-4.71	3.14	365.3	Not Acceptable

Product: Total Cyanide **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Cyanide	WP	71	0.254	0.57	0.39-0.73	5.37	335.3	Not Acceptable

Analytical Products Group, Inc.

PERFORMANCE REPORT

WP Performance Summary

April 2001

APG Customer Code: 3655

Analytical Management Lab
Suite B
15130 South Keeler
Olathe, KS 66062

Laboratory of EXCELLENCE

NVLAP®
LAB CODE 200384-0

Water Pollution (WP) Proficiency Testing Program Report

Introduction:

The Analytical Products Group, Inc. Proficiency Testing Program has been designed to evaluate your laboratory's performance against other participating laboratories on the same set of standards. This report has been specifically prepared for your organization to assist you in ensuring that your procedures are in-control. Therefore, in most cases sample levels are not designed to challenge the detection limits of the method.

The Analytical Products Group Proficiency Testing Program is certified to meet all of the following requirements:

- Accredited by NIST according to the National Voluntary Laboratory Accreditation Program (Handbook 150-19) for Chemical Calibrations: Providers of Proficiency Testing (NVLAB Code 200384-0)
- Analytical Products Group, Inc. Quality System is registered with DNVCertification to meet the ISO 9001 standard to design, manufacture, and distribute chemical reference materials and operate Proficiency Testing Programs (Certificate # 98-HOU-AQ-9513)
- Proficiency Testing Program is certified to meet all requirements as specified in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).
- Certified to meet ISO/REMCO Harmonized Protocol for Proficiency Testing of Analytical Laboratories
- Certified to meet ASTM E1301

Report Layout

This report contains the following sub-reports:

Introduction -This section is the general introduction to the Analytical Products Group, Inc. Proficiency Testing Program as well as all of the certifications that the organization maintains.

Terms & Definitions - Defines all of the terms used throughout the report. This is an excellent section to refer to first if you have any questions.

Laboratory Benchmark Report - This report presents an overall summary of the study as well as your performance relative to all other study participants.

Comment Report - Contains comments submitted to Analytical Products Group by the program participants or comments by Analytical Products Group to program participants concerning unusual characteristics of the data set or problems with standards which could influence the interpretation of the data. Please review this section thoroughly before examining your data.

Exception Report - This report is a summary of all of the analytes that resulted in an Evaluation of "Not Acceptable." The Exception Report is designed to very quickly identify for your laboratory the areas that may require your immediate attention.

Performance Evaluation Summary Report - Provides a summary of all of your data for each analyte. This summary is formatted similar to the US EPA DMR-QA Reports.

Detail Report- This report lists all the details of your data. Also included in this section are useful graphs indicating the APG Assigned Value, the EPA Mean, the EPA Acceptance Limits and your Reported Value. Another graph included in this section shows your Z score for the analyte of interest.

The goal of this report is to provide your laboratory with the information you need to monitor and improve your quality. If you have any questions or feedback, please give us a call (800-272-4442). Thank you for choosing Analytical Products Group as your proficiency testing provider.

Sincerely,

Thomas J. Coyner

Thomas J. Coyner
Proficiency Program Manager

Terms & Definitions

Average Acceptable Data - This value is the average percent of data that is within the EPA Acceptance Limits for this study. The Average Acceptable Data is reported for all laboratories as a whole as well as your laboratory as an individual. These values can be found in the Laboratory Benchmark Report.

Average Percentile for All Parameters - This is your laboratory's rank on each parameter and the related rank for that parameter. The average percentile is the average of this data across all parameters reported. It is a different measure of performance than percent acceptable data since it is affected by your relative performance on each parameter. The report provides an average for this measure for all laboratories reporting as well as your laboratory's performance.

Analyte - Name of the analyte of interest as found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) or NELAC Field of Testing List.

Analyte Code - This value is the code given to the analyte by the US EPA. These codes can be found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045). If no code is listed, then this analyte is not part of the US EPA program.

APG Assigned Value - This value is the calculated True Value of the standard based upon the actual composition of the standard.

APG Certified Value - This value is the result of the quality control testing performed on the sample by Analytical Products Group before the sample is shipped to the customer. Through this testing, the samples are traced against a NIST Standard Reference Material where available or an external standard if a NIST Standard Reference Material is not available. The reported uncertainty of this value is expressed as two times the standard deviation (s) divided by the square root of the number of samples (n) tested (i.e. 2s/vn). This quality control testing meets all of the requirements as specified by the NIST National Voluntary Laboratory Accreditation Program (NVLAP) Handbook 150-19 (Chemical Calibration: Providers of Proficiency Testing).

EPA Acceptance Range - This range is calculated based upon the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045). For the Water Pollution Program (WP), EPA Acceptance Limits are defined as ± three (3) EPA Standard Deviations from the EPA Mean.

EPA Mean - If the analyte is included in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then this value is the Mean Value calculated per the Criteria Document. If the analyte is not included in the Criteria Document, then this value is the Study Mean.

Terms & Definitions (continued)

Evaluation - This value is determined by the EPA Acceptance Limits and the Reported Value. Evaluation may have one of following values:

Value	Description
Acceptable	The Reported Value was within the EPA Acceptable Limits or a less than (<) value was reported AND zero (0) was included in the EPA Acceptance Limit Range AND the numeric part of the Reported Value was less than the upper limit of the EPA Acceptance Limit.
Not Acceptable	The Reported Value is outside of the EPA Acceptance Limits or a less than (<) value was reported AND zero (0) is not included in the EPA Acceptance Limits range or the numeric part of the Reported Value was greater than the upper limit of the EPA Acceptance Limit or a greater than value (>) was reported.
Check for Error	The Reported Value is between two (2) and three (3) EPA Standard Deviations from the EPA Mean.
Unusable	The Reported Value was a text value which could not be evaluated.
Blank	No data was reported for this analyte or the analyte was not spiked.

Inter-Laboratory Statistics - This data is a summary of all of the data from all study participants for the analyte of interest. Inter-Laboratory Statistics are only reported if a minimum number of laboratories analyzed and reported data for the analyte of interest.

Laboratory Pass Percentage - This is the percentage of study participants that reported values within the EPA Acceptance Limits for the analyte of interest.

Number of Acceptable Data Points - This value is the number of data points reported by your laboratory that were within the EPA Acceptance Limits.

Program - This value indicates which program the analyte of interest is a part. For Water Pollution Studies (WP), the only values that Program will have are 'WP' or 'APG+'. If the analyte and range are consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then the Program value will have a value of 'WP' and if this is not the case then the Program value will be 'APG+', indicating that the analyte and/or range was specified by Analytical Products Group.

Rank on Percent Acceptable Data - This is your laboratories rank out of all possible ranks in the current study based on percent acceptable data. It is important to note that several laboratories can achieve the same rank.

Rank on Average Percentile Data - This value is your laboratories rank out of all possible ranks based on Your Ranking converted to a percentile. It is important to note that several laboratories can achieve the same rank.

Reported Value - This value is the result that was reported by your organization for the particular analyte. Every effort has been made to ensure correct data entry. Should you find a data entry error, please notify Analytical Products Group, Inc. and we will correct the error and re-issue the report. If the Reported Value is reported as a less than (<) than figure, then the only way that the Evaluation will result in an "Acceptable" value is if the numeric part of the Reported Value (i.e. 8 if the Report Value were <8) is less then the upper limit of the EPA Acceptance Limit AND zero (0) is included in the EPA Acceptance Limit. Any other non-numeric value (i.e. BDL, ND, >10, etc.) cannot be evaluated and will return an Evaluation result of "Unusable." This method of evaluation is consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).

Solids Standards - Solids standards are individually prepared, therefore, each standard has a unique APG Assigned Value.

Study Mean - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Mean is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Terms & Definitions (continued)

Study Standard Deviation - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Standard Deviation is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Weighted Rank on Percent Acceptable Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percent Acceptable Data is the weighted rank of your laboratory based on Percent Acceptable Data and the number of analytes reported.

Weight Rank on Percentile Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percentile Data is the weighted rank of your laboratory based on Rank on Average Percentile Data and the number of analytes reported.

Your Ranking - This is your ranking based on your Z Score for the analyte of interest among all possible rankings. It is important to note that several laboratories can achieve the same rank.

Z-Score - This figure corresponds to how far your Reported Value was from the EPA Mean, reported in terms of EPA Standard Deviation units. As a point of reference, the US EPA Acceptance Limits are plus or minus three (3) EPA Standard Deviations from the EPA Mean.

Laboratory Benchmark Report

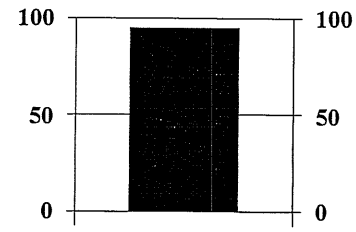
Customer Code: 3655 **WP April 2001**

Summary of Ranking Data

Summary of National Ranking Data

Total Number of Laboratories Reporting:	191
Average Percent Acceptable Data:	94.92%
Average Percentile for all Parameters:	62.01%

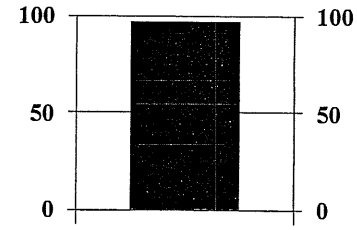
National Avg % Acceptable
94.9 %



Summary of Laboratory Ranking:

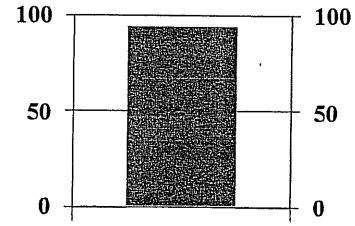
Total Data Points Reported:	177
Number of Acceptable Data Points:	171
Percent Acceptable Data:	96.61%
Average Percentile for all Parameters:	61.95%

Laboratory Avg % Acceptable
96.61%



Rank on Percent Acceptable Data:	25 of 65
Rank on Average Percentile Data:	115 of 173
Weighted Rank on Percent Acceptable Data:	13 of 90
Weighted Rank on Average Percentile Data:	12 of 185

Weighted Rank Avg Percentile
93.5 %



Comment Report

There are no comments to report.

Exception Report

WP April 2001
Study Closing Date 05/15/2001

Product: Nutrient **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Ammonia Nitrogen as N	WP	31	9.99	13.1	10.2-15.9	3.17	EPA350.1	Not Acceptable

Product: PCB's in Oil **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aroclor 1016/1242 in Oil Sample 2	WP	99	42.2	20	2.29-29.9	5.67	EPA608	Not Acceptable

Product: Total Cyanide **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Cyanide	WP	71	0.494	0.38	0.26-0.49	2.92	EPA335.2	Not Acceptable

Product: Trace Metals **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Cadmium	WP	4	138	118	100-135	3.41	EPA200.7	Not Acceptable

Product: Volatiles **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Chlorobenzene	WP	64	<2.0	33.6	23.9-42.7		EPA624	Not Acceptable

Performance Summary

WP April 2001
Study Closing Date 05/15/2001

Product: Acid Extractables **WP Lot Number: 30714** **APG+ Lot Number: 30198**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
2,4,5-Trichlorophenol	WP		52.6	70.7	30-89.5	0.72	EPA625	Acceptable
2,4,6-Trichlorophenol	WP		24.3	32.8	12.2-42.3	0.6	EPA625	Acceptable
2,4-Dichlorophenol	WP		39.4	55.4	20.1-68.6	0.61	EPA625	Acceptable
2,4-Dimethylphenol	WP		36.6	52.9	9.22-70.7	0.33	EPA625	Acceptable
2,4-Dinitrophenol	WP		<10				EPA625	Acceptable
2-Chlorophenol	WP		115	177	49.5-221	0.7	EPA625	Acceptable
2-Methyl-4,6-Dinitrophenol	WP		<10				EPA625	Acceptable
2-Nitrophenol	WP		19.7	30.2	9.92-38.8	0.98	EPA625	Acceptable
4-Chloro-3-methylphenol	WP		100	134	52.6-172	0.6	EPA625	Acceptable
4-Nitrophenol	WP		17.8	48.5	0-72.2	0.68	EPA625	Acceptable
Pentachlorophenol	WP		69.5	100	25.3-138	0.65	EPA625	Acceptable
Phenol	WP		16.8	43.6	0-61	0.67	EPA625	Acceptable

Product: Aluminum High Level **WP Lot Number: 30175** **APG+ Lot Number: 30192**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aluminum High Level	WP		0.778	0.75	0.64-0.83	1.15	EPA200.7	Acceptable

Performance Summary

WP April 2001
Study Closing Date 05/15/2001

Product: Base Neutral Extractables **WP Lot Number: 30716** **APG+ Lot Number: 30199**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
1,2,4-Trichlorobenzene	WP		83.1	169	49.6-198	1.65	EPA625	Acceptable
1,2-Dichlorobenzene	WP		20.2	41.3	7.5-57.8	1.49	EPA625	Acceptable
1,3-Dichlorobenzene	WP		31.7	70.9	12.4-89.8	1.5	EPA625	Acceptable
1,4-Dichlorobenzene	WP		15.4	33.6	6.73-44.7	1.63	EPA625	Acceptable
2,4-Dinitrotoluene (2,4-DNT)	WP		20.4	28.8	7.72-37.3	0.43	EPA625	Acceptable
2,6-Dinitrotoluene (2,6-DNT)	WP		23.5	32.6	12.4-40	0.59	EPA625	Acceptable
2-Chloronaphthalene	WP		99.0	162	65.4-181	1.24	EPA625	Acceptable
3,3'-Dichlorobenzidine	WP		<10				EPA625	Acceptable
4-Bromophenyl phenyl ether	WP		<10				EPA625	Acceptable
4-Chlorophenyl phenyl ether	WP		<10				EPA625	Acceptable
Acenaphthene	WP		31.6	46.3	18.2-59.2	1.04	EPA625	Acceptable
Acenaphthylene	WP		12.2	14.1	4.62-19.2	0.12	EPA625	Acceptable
Anthracene	WP		71.6	94.4	34.1-122	0.44	EPA625	Acceptable
Benzo(a)anthracene	WP		37.4	49.2	26.9-58.7	1.02	EPA625	Acceptable
Benzo(a)pyrene	WP		<10				EPA625	Acceptable
Benzo(b)fluoranthene	WP		<10				EPA625	Acceptable
Benzo(g,h,i)perylene	WP		<10				EPA625	Acceptable
Benzo(k)fluoranthene	WP		<10				EPA625	Acceptable
Bis(2-Chloroethoxy)methane	WP		<10				EPA625	Acceptable
Bis(2-Chloroethyl)ether	WP		<10				EPA625	Acceptable
Bis(2-Chloroisopropyl)ether	WP		<10				EPA625	Acceptable
Bis(2-Ethylhexyl)phthalate	WP		56.1	71.5	17.5-104	0.32	EPA625	Acceptable
Butyl benzyl phthalate	WP		81.9	106	0-191	0.16	EPA625	Acceptable

Performance Summary

WP April 2001
Study Closing Date 05/15/2001

Product: Base Neutral Extractables **WP Lot Number: 30716** **APG+ Lot Number: 30199**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Chrysene	WP		42.7	56.6	23.2-73.8	0.69	EPA625	Acceptable
Dibenzo(a,h)anthracene	WP		<10				EPA625	Acceptable
Diethyl phthalate	WP		39.9	54.3	7.38-82.4	0.4	EPA625	Acceptable
Dimethyl phthalate	WP		78.2	121	0-185	0.011	EPA625	Acceptable
Di-n-butyl phthalate	WP		20.7	24.8	13.7-36.9	1.19	EPA625	Acceptable
Di-n-octyl phthalate	WP		65.3	87.1	14-127	0.29	EPA625	Acceptable
Fluoranthene	WP		92.3	120	51.2-155	0.62	EPA625	Acceptable
Fluorene	WP		20.9	28.6	12.2-37.7	0.97	EPA625	Acceptable
Hexachlorobenzene	WP		18.0	23.9	10.4-31.3	0.8	EPA625	Acceptable
Hexachlorobutadiene	WP		47.0	105	17.7-126	1.37	EPA625	Acceptable
Hexachlorocyclopentadiene	WP		36.4	61.3	0-80.6	0.034	EPA625	Acceptable
Hexachloroethane	WP		21.5	52.5	6.6-65.1	1.48	EPA625	Acceptable
Indeno(1,2,3-cd)pyrene	WP		<10				EPA625	Acceptable
Isophorone	WP		124	140	47.1-187	0.3	EPA625	Acceptable
Naphthalene	WP		83.3	145	34.9-189	1.12	EPA625	Acceptable
Nitrobenzene (NB)	WP		51.5	80.5	26.8-105	1.11	EPA625	Acceptable
N-nitrosodimethylamine	WP		<10				EPA625	Acceptable
N-nitrosodi-n-propylamine	WP		<10				EPA625	Acceptable
Phenanthrene	WP		74.1	104	38.3-136	0.8	EPA625	Acceptable
Pyrene	WP		35.6	47	15.3-67	0.65	EPA625	Acceptable

Performance Summary

WP April 2001
Study Closing Date 05/15/2001

Product: BTEX **WP Lot Number: 30169** **APG+ Lot Number: 30202**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Benzene	WP	65	51.6	52	37.5-66.9	0.12	EPA624	Acceptable
Ethylbenzene	WP	66	45.3	39.5	26.4-51.5	1.5	EPA624	Acceptable
m-Xylene / p-Xylene	WP		<10				EPA624	Acceptable
o-Xylene	WP		168	161	115-221	0	EPA624	Acceptable
Toluene	WP	67	64.7	60.9	43.9-75.9	0.9	EPA624	Acceptable
Total Xylenes	WP		168	161	92.4-216	0.68	EPA624	Acceptable

Product: Chlordane (Total) **WP Lot Number: 30163** **APG+ Lot Number: 30209**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Chlordane (Total)	WP	53	4.34	5.36	2.34-7.62	0.73	EPA608	Acceptable

Product: Demand **WP Lot Number: 30146** **APG+ Lot Number: 30180**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Biochemical Oxygen Demand	WP	38	93.9	101	51-151	0.42	EPA405.1	Acceptable
Carbonaceous BOD	WP	102		86.8	38.9-135			
Chemical Oxygen Demand	WP	36	171.4	163	125-186	1.52	EPA410.1	Acceptable
Total Organic Carbon	WP	37		64.5	53.9-74.2			

Performance Summary

WP April 2001
Study Closing Date 05/15/2001

Product: Fluoride **WP Lot Number: 30176** **APG+ Lot Number: 30193**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Fluoride	WP	29	1.10	1.12	0.96-1.28	0.38	EPA300.0	Acceptable

Product: Hexavalent Chromium **WP Lot Number: 30165** **APG+ Lot Number: 30195**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Chromium, Hexavalent	WP		42.8	41.7	28-53.9	0.44	EPA218.5	Acceptable

Product: Minerals **WP Lot Number: 30149-30150** **APG+ Lot Number: 30184-30185**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Alkalinity as CaCO3	WP	27	28.6	31.8	27.3-37.2	2.18	EPA310.1	Check for Error
Calcium	WP	23	18.5	17	14.9-19.6	1.54	EPA200.7	Acceptable
Chloride	WP	28	97.9	105	95.5-115	2.23	EPA300.0	Check for Error
Conductivity	WP	20	485	495	441-517	0.47	EPA120.1	Acceptable
Magnesium	WP	24	8.94	8.98	7.8-10.1	0.077	EPA200.7	Acceptable
Potassium	WP	26	18.0	17	14.6-19.6	1.08	EPA200.7	Acceptable
Sodium	WP	25	49.1	47	42.3-51.9	1.25	EPA200.7	Acceptable
Sulfate	WP	30	21.0	22.5	17.6-26.8	0.79	EPA300.0	Acceptable
Total Dissolved Solids	WP	21	252	262	186-316	0.046	EPA160.1	Acceptable
Total Hardness as CaCO3	WP	22	83.1	79.6	71-88.8	1.08	STDMTH2340B	Acceptable

Performance Summary

WP April 2001
Study Closing Date 05/15/2001

Product: Nutrient **WP Lot Number: 30147-30148** **APG+ Lot Number: 30181-30182**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Ammonia Nitrogen as N	WP	31	9.99	13.1	10.2-15.9	3.17	EPA350.1	**Not Acceptable**
Nitrate Nitrogen as N	WP	32	24.3	26.9	21.4-31.9	1.31	EPA300.0	Acceptable
Orthophosphate as P	WP	33	3.66	4.03	3.44-4.66	1.95	EPA300.0	Acceptable
Total Kjeldahl Nitrogen	WP	34		0.7	0.17-1.36			
Total Phosphorus as P	WP	35	2.95	3	2.28-3.54	0.19	EPA365.3	Acceptable

Product: Oil & Grease **WP Lot Number: 30157** **APG+ Lot Number: 30183**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Oil & Grease	WP	104	32.2	36.9	24.1-42.5	0.36	EPA413.1	Acceptable

Product: PCB's in Oil **WP Lot Number: 30160-30161**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aroclor 1016/1242 in Oil Sample 1	WP	99	<15				EPA608	Acceptable
Aroclor 1016/1242 in Oil Sample 2	WP	99	42.2	20	2.29-29.9	5.67	EPA608	**Not Acceptable**
Aroclor 1254 in Oil Sample 1	WP	100	<15				EPA608	Acceptable
Aroclor 1254 in Oil Sample 2	WP	100	<15				EPA608	Acceptable
Aroclor 1260 in Oil Sample 1	WP	101	38.9	43	7.79-60.5	0.54	EPA608	Acceptable
Aroclor 1260 in Oil Sample 2	WP	101	<15				EPA608	Acceptable

Performance Summary

WP April 2001
Study Closing Date 05/15/2001

Product: PCB's in Water **WP Lot Number: 30159-30589** **APG+ Lot Number: 30206**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aroclor 1016/1242 Sample 1	WP	40	<1.0				EPA608	Acceptable
Aroclor 1016/1242 Sample 2	WP	40	<1.0				EPA608	Acceptable
Aroclor 1221 Sample 1	WP		<1.0				EPA608	Acceptable
Aroclor 1221 Sample 2	WP		<1.0				EPA608	Acceptable
Aroclor 1232 Sample 1	WP	42	<1.0				EPA608	Acceptable
Aroclor 1232 Sample 2	WP	42	<1.0				EPA608	Acceptable
Aroclor 1248 Sample 1	WP	44	<1.0				EPA608	Acceptable
Aroclor 1248 Sample 2	WP	44	<1.0				EPA608	Acceptable
Aroclor 1254 Sample 1	WP	45	4.74	4.56	2.14-5.96	1.08	EPA608	Acceptable
Aroclor 1254 Sample 2	WP	45	<1.0				EPA608	Acceptable
Aroclor 1260 Sample 1	WP	46	<1.0				EPA608	Acceptable
Aroclor 1260 Sample 2	WP	46	3.56	3.58	1.85-4.7	0.62	EPA608	Acceptable

Performance Summary

WP April 2001
Study Closing Date 05/15/2001

Product: Pesticides **WP Lot Number: 30715** **APG+ Lot Number: 30200**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
4,4'-DDD	WP	49	3.68	3.61	1.77-5.03	0.52	EPA608	Acceptable
4,4'-DDE	WP	50	2.16	2.17	1.03-2.97	0.5	EPA608	Acceptable
4,4'-DDT	WP	51	6.00	6.84	3.14-9.25	0.19	EPA608	Acceptable
Aldrin	WP	47	0.56	0.61	0.15-0.83	0.64	EPA608	Acceptable
alpha-BHC	WP		5.55	6.07	2.27-8.39	0.22	EPA608	Acceptable
beta-BHC	WP		9.82	10.5	3.94-15.1	0.16	EPA608	Acceptable
delta-BHC	WP		34.3	36.8	8-53.4	0.48	EPA608	Acceptable
Dieldrin	WP	48	2.20	2.26	1.17-3.13	0.15	EPA608	Acceptable
Endosulfan I	WP		0.38	0.32	0.075-0.59	0.59	EPA608	Acceptable
Endosulfan II	WP		23.4	30.8	5.88-49.6	0.59	EPA608	Acceptable
Endosulfan sulfate	WP		10.4	12.2	2.5-19.8	0.24	EPA608	Acceptable
Endrin	WP		<0.1				EPA608	Acceptable
Endrin aldehyde	WP		<0.1				EPA608	Acceptable
gamma-BHC	WP		8.6	9.24	3.03-13.4	0.24	EPA608	Acceptable
Heptachlor	WP	52	2.32	2.5	0.66-3.49	0.51	EPA608	Acceptable
Heptachlor epoxide (beta)	WP	78	1.62	1.72	0.87-2.21	0.36	EPA608	Acceptable
Methoxychlor	WP		7.72	8.23	3.24-12.1	0.027	EPA608	Acceptable

Product: pH **WP Lot Number: 30151** **APG+ Lot Number: 30186**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
pH	WP	19	7.63	7.64	7.43-7.85	0.14	EPA150.1	Acceptable

Performance Summary

WP April 2001
Study Closing Date 05/15/2001

Product: Polynuclear Aromatic Hydrocarbons WP Lot Number: 30168 APG+ Lot Number: 30201

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Benzo(a)anthracene	WP			1.52	0.53-1.96			
Chrysene	WP			1.22	0.32-1.72			
Fluoranthene	WP			1.76	0.88-2.2			
Fluorene	WP			22	14.8-19.1			
Naphthalene	WP			20	8.84-21.8			
Phenanthrene	WP			0.55	0.43-0.77			
Pyrene	WP			0.44	0.13-0.69			

Product: Solids WP Lot Number: 66657

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Dissolved Solids	WP		130	125	52.2-226	0.31	EPA160.1	Acceptable
Total Solids	WP		166	163	123-215	0.2	EPA160.3	Acceptable
Total Suspended Solids	WP	72	36	38.5	28.5-41.1	0.57	EPA160.2	Acceptable

Product: Total Cyanide WP Lot Number: 30155 APG+ Lot Number: 30190

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Cyanide	WP	71	0.494	0.38	0.26-0.49	2.92	EPA335.2	**Not Acceptable**

Performance Summary

WP April 2001
Study Closing Date 05/15/2001

Product: Total Phenolics **WP Lot Number: 30154** **APG+ Lot Number: 30189**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Phenolics	WP	97	0.359	0.65	0.23-0.63	1.08	EPA420.2	Acceptable

Product: Total Residual Chlorine **WP Lot Number: 30156** **APG+ Lot Number: 30191**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Residual Chlorine	WP	98	0.96	0.96	0.71-1.15	0.42	EPA330.4	Acceptable

Product: Toxaphene **WP Lot Number: 30172** **APG+ Lot Number: 30207**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Toxaphene	WP		11.2	8.91	0-15.9	1.36	EPA608	Acceptable

Product: TPH 418.1 **WP Lot Number: 30179** **APG+ Lot Number: 30205**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
TPH by 418.1	WP			10.2	1.01-24.3			

Product: TPH Diesel (DRO) **WP Lot Number: 30170** **APG+ Lot Number: 30204**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
TPH Diesel (DRO)	WP		2.82	3.21	1.02-4.08	0.53	OA-2	Acceptable

Performance Summary

WP April 2001

Study Closing Date 05/15/2001

Product: TPH Gasoline (GRO) **WP Lot Number: 30171** **APG+ Lot Number: 30203**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
TPH Gasoline (GRO)	WP		2.86	3.92	1.3-4.31	0.1	OA-1	Acceptable

Performance Summary

WP April 2001
Study Closing Date 05/15/2001

Product: Trace Metals **WP Lot Number: 30152-30153** **APG+ Lot Number: 30187-30188**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aluminum	WP	1	1550	1500	1290-1710	0.71	EPA200.7	Acceptable
Antimony	WP	16	151	138	87-171	1.57	EPA200.7	Acceptable
Arsenic	WP	2	594	574	481-672	0.54	EPA200.7	Acceptable
Barium	WP		1350	1310	1120-1490	0.81	EPA200.7	Acceptable
Beryllium	WP	3	356	376	320-425	0.91	EPA200.7	Acceptable
Boron	WP			263	222-314			
Cadmium	WP	4	138	118	100-135	3.41	EPA200.7	**Not Acceptable**
Chromium	WP	6	126	111	94.8-128	2.74	EPA200.7	**Check for Error**
Cobalt	WP	5	413	375	329-421	2.48	EPA200.7	**Check for Error**
Copper	WP	7	603	585	531-642	0.92	EPA200.7	Acceptable
Iron	WP	8	1880	1750	1550-1970	1.71	EPA200.7	Acceptable
Lead	WP	12	237	212	181-244	2.38	EPA200.7	**Check for Error**
Manganese	WP	10	790	750	673-833	1.39	EPA200.7	Acceptable
Mercury	WP	9	2.18	2.18	1.52-2.83	0	EPA245.1	Acceptable
Molybdenum	WP	74	417	396	339-453	1.11	EPA200.7	Acceptable
Nickel	WP	11	676	631	568-706	1.7	EPA200.7	Acceptable
Selenium	WP	13	248	226	176-262	2.01	EPA200.7	**Check for Error**
Silver	WP	17	225	210	179-240	1.49	EPA200.7	Acceptable
Strontium	WP	75		74.5	63.1-85.8			
Thallium	WP	18	424	403	324-469	1.16	EPA200.7	Acceptable
Titanium	WP	76		121	103-137			
Vanadium	WP	14	258	251	224-275	1.06	EPA200.7	Acceptable
Zinc	WP	15	552	500	441-565	2.38	EPA200.7	**Check for Error**

Performance Summary

WP April 2001
Study Closing Date 05/15/2001

Product: Volatiles **WP Lot Number: 30164** **APG+ Lot Number: 30197**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
1,1,1-Trichloroethane	WP	56	48.1	46.3	30-61.1	0.48	EPA624	Acceptable
1,1,2,2-Tetrachloroethane	WP		<2.0				EPA624	Acceptable
1,1,2-Trichloroethane	WP		170	168	113-218	0.23	EPA624	Acceptable
1,1-Dichloroethane	WP		<2.0				EPA624	Acceptable
1,1-Dichloroethene	WP		<2.0				EPA624	Acceptable
1,2-Dichlorobenzene	WP	94	40.5	40	28.2-50.7	0.27	EPA624	Acceptable
1,2-Dichloroethane	WP	54	57.8	58.1	40.1-77.3	0.15	EPA624	Acceptable
1,2-Dichloropropane	WP		<2.0				EPA624	Acceptable
1,3-Dichlorobenzene	WP	96	48.3	48	33.9-59.8	0.33	EPA624	Acceptable
1,4-Dichlorobenzene	WP	95	44.8	44.1	30.3-56.9	0.27	EPA624	Acceptable
Benzene	WP	65	18.4	19.2	13.6-25.1	0.47	EPA624	Acceptable
Bromodichloromethane	WP	60	15.9	15.8	10.9-20.7	0.061	EPA624	Acceptable
Bromoform	WP	62	25.1	28.7	18.2-39.4	1.04	EPA624	Acceptable
Bromomethane	WP		<2.0				EPA624	Acceptable
Carbon tetrachloride	WP	58	19.5	19	12-26.9	0.04	EPA624	Acceptable
Chlorobenzene	WP	64	<2.0	33.6	23.9-42.7		EPA624	**Not Acceptable**
Chloroethane	WP		<2.0				EPA624	Acceptable
Chloroform	WP	55	35.2	34.1	23.8-43.8	0.42	EPA624	Acceptable
Chloromethane	WP		<2.0				EPA624	Acceptable
cis-1,3-Dichloropropene	WP		<2.0				EPA624	Acceptable
Dibromochloromethane	WP	61	41.9	39.9	26.4-53	0.5	EPA624	Acceptable
Ethylbenzene	WP	66	20.2	18.7	12.7-24.2	0.94	EPA624	Acceptable
Methylene chloride	WP	63	54.6	58.7	37.1-80.7	0.59	EPA624	Acceptable

APG Customer 3655 Analytical Management Lab
EPA Lab Code KS00902 15130 South Keeler
Olathe, KS 66062

Performance Summary

WP April 2001
Study Closing Date 05/15/2001

Product: Volatiles **WP Lot Number: 30164** **APG+ Lot Number: 30197**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Tetrachloroethene	WP	59	19.9	19.2	12.6-25	0.54	EPA624	Acceptable
Toluene	WP	67	39.2	38.2	27.4-47.9	0.47	EPA624	Acceptable
trans-1,2-Dichloroethene	WP		161	175	113-238	0.72	EPA624	Acceptable
trans-1,3-Dichloropropene	WP		<2.0				EPA624	Acceptable
Trichloroethene	WP	57	52.2	51.3	33.3-66.3	0.44	EPA624	Acceptable
Trichlorofluoromethane	WP		<2.0				EPA624	Acceptable
Vinyl Chloride	WP		<2.0				EPA624	Acceptable

Analytical Products Group, Inc.

PERFORMANCE REPORT

WP Performance Summary

October 2001

APG Customer Code: 3655

Analytical Management Lab
Suite B
15130 South Keeler
Olathe, KS 66062

Laboratory of
EXCELLENCE

NVLAP®
LAB CODE 200384-0

Water Pollution (WP) Proficiency Testing Program Report

Introduction:

The Analytical Products Group, Inc. Proficiency Testing Program has been designed to evaluate your laboratory's performance against other participating laboratories on the same set of standards. This report has been specifically prepared for your organization to assist you in ensuring that your procedures are in-control. Therefore, in most cases sample levels are not designed to challenge the detection limits of the method.

The Analytical Products Group Proficiency Testing Program is certified to meet all of the following requirements:

- Accredited by NIST according to the National Voluntary Laboratory Accreditation Program (Handbook 150-19) for Chemical Calibrations: Providers of Proficiency Testing (NVLAB Code 200384-0)
- Analytical Products Group, Inc. Quality System is registered with DNVCertification to meet the ISO 9001 standard to design, manufacture, and distribute chemical reference materials and operate Proficiency Testing Programs (Certificate # 98-HOU-AQ-9513)
- Proficiency Testing Program is certified to meet all requirements as specified in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).
- Certified to meet ISO/REMCO Harmonized Protocol for Proficiency Testing of Analytical Laboratories
- Certified to meet ASTM E1301

Report Layout

This report contains the following sub-reports:

Introduction -This section is the general introduction to the Analytical Products Group, Inc. Proficiency Testing Program as well as all of the certifications that the organization maintains.

Terms & Definitions - Defines all of the terms used throughout the report. This is an excellent section to refer to first if you have any questions.

Laboratory Benchmark Report - This report presents an overall summary of the study as well as your performance relative to all other study participants.

Comment Report - Contains comments submitted to Analytical Products Group by the program participants or comments by Analytical Products Group to program participants concerning unusual characteristics of the data set or problems with standards which could influence the interpretation of the data. Please review this section thoroughly before examining your data.

Exception Report - This report is a summary of all of the analytes that resulted in an Evaluation of "Not Acceptable." The Exception Report is designed to very quickly identify for your laboratory the areas that may require your immediate attention.

Performance Evaluation Summary Report - Provides a summary of all of your data for each analyte. This summary is formatted similar to the US EPA DMR-QA Reports.

Detail Report- This report lists all the details of your data. Also included in this section are useful graphs indicating the APG Assigned Value, the EPA Mean, the EPA Acceptance Limits and your Reported Value. Another graph included in this section shows your Z score for the analyte of interest.

The goal of this report is to provide your laboratory with the information you need to monitor and improve your quality. If you have any questions or feedback, please give us a call (800-272-4442). Thank you for choosing Analytical Products Group as your proficiency testing provider.

Sincerely,

Thomas J. Coyner

Thomas J. Coyner
Proficiency Program Manager

Terms & Definitions

Average Acceptable Data - This value is the average percent of data that is within the EPA Acceptance Limits for this study. The Average Acceptable Data is reported for all laboratories as a whole as well as your laboratory as an individual. These values can be found in the Laboratory Benchmark Report.

Average Percentile for All Parameters - This is your laboratory's rank on each parameter and the related rank for that parameter. The average percentile is the average of this data across all parameters reported. It is a different measure of performance than percent acceptable data since it is affected by your relative performance on each parameter. The report provides an average for this measure for all laboratories reporting as well as your laboratory's performance.

Analyte - Name of the analyte of interest as found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) or NELAC Field of Testing List.

Analyte Code - This value is the code given to the analyte by the US EPA. These codes can be found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045). If no code is listed, then this analyte is not part of the US EPA program.

APG Assigned Value - This value is the calculated True Value of the standard based upon the actual composition of the standard.

APG Certified Value - This value is the result of the quality control testing performed on the sample by Analytical Products Group before the sample is shipped to the customer. Through this testing, the samples are traced against a NIST Standard Reference Material where available or an external standard if a NIST Standard Reference Material is not available. The reported uncertainty of this value is expressed as two times the standard deviation (s) divided by the square root of the number of samples (n) tested (i.e. 2s/vn). This quality control testing meets all of the requirements as specified by the NIST National Voluntary Laboratory Accreditation Program (NVLAP) Handbook 150-19 (Chemical Calibration: Providers of Proficiency Testing).

EPA Acceptance Range - This range is calculated based upon the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045). For the Water Pollution Program (WP), EPA Acceptance Limits are defined as ± three (3) EPA Standard Deviations from the EPA Mean.

EPA Mean - If the analyte is included in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then this value is the Mean Value calculated per the Criteria Document. If the analyte is not included in the Criteria Document, then this value is the Study Mean.

Terms & Definitions (continued)

Evaluation - This value is determined by the EPA Acceptance Limits and the Reported Value. Evaluation may have one of following values:

Value	Description
Acceptable	The Reported Value was within the EPA Acceptable Limits or a less than (<) value was reported AND zero (0) was included in the EPA Acceptance Limit Range AND the numeric part of the Reported Value was less than the upper limit of the EPA Acceptance Limit.
Not Acceptable	The Reported Value is outside of the EPA Acceptance Limits or a less than (<) value was reported AND zero (0) is not included in the EPA Acceptance Limits range or the numeric part of the Reported Value was greater than the upper limit of the EPA Acceptance Limit or a greater than value (>) was reported.
Check for Error	The Reported Value is between two (2) and three (3) EPA Standard Deviations from the EPA Mean.
Unusable	The Reported Value was a text value which could not be evaluated.
Blank	No data was reported for this analyte or the analyte was not spiked.

Inter-Laboratory Statistics - This data is a summary of all of the data from all study participants for the analyte of interest. Inter-Laboratory Statistics are only reported if a minimum number of laboratories analyzed and reported data for the analyte of interest.

Laboratory Pass Percentage - This is the percentage of study participants that reported values within the EPA Acceptance Limits for the analyte of interest.

Number of Acceptable Data Points - This value is the number of data points reported by your laboratory that were within the EPA Acceptance Limits.

Program - This value indicates which program the analyte of interest is a part. For Water Pollution Studies (WP), the only values that Program will have are 'WP' or 'APG+'. If the analyte and range are consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then the Program value will have a value of 'WP' and if this is not the case then the Program value will be 'APG+', indicating that the analyte and/or range was specified by Analytical Products Group.

Rank on Percent Acceptable Data - This is your laboratories rank out of all possible ranks in the current study based on percent acceptable data. It is important to note that several laboratories can achieve the same rank.

Rank on Average Percentile Data - This value is your laboratories rank out of all possible ranks based on Your Ranking converted to a percentile. It is important to note that several laboratories can achieve the same rank.

Reported Value - This value is the result that was reported by your organization for the particular analyte. Every effort has been made to ensure correct data entry. Should you find a data entry error, please notify Analytical Products Group, Inc. and we will correct the error and re-issue the report. If the Reported Value is reported as a less than (<) than figure, then the only way that the Evaluation will result in an "Acceptable" value is if the numeric part of the Reported Value (i.e. 8 if the Report Value were <8) is less then the upper limit of the EPA Acceptance Limit AND zero (0) is included in the EPA Acceptance Limit. Any other non-numeric value (i.e. BDL, ND, >10, etc.) cannot be evaluated and will return an Evaluation result of "Unusable." This method of evaluation is consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).

Solids Standards - Solids standards are individually prepared, therefore, each standard has a unique APG Assigned Value.

Study Mean - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Mean is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Terms & Definitions (continued)

Study Standard Deviation - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Standard Deviation is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Weighted Rank on Percent Acceptable Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percent Acceptable Data is the weighted rank of your laboratory based on Percent Acceptable Data and the number of analytes reported.

Weight Rank on Percentile Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percentile Data is the weighted rank of your laboratory based on Rank on Average Percentile Data and the number of analytes reported.

Your Ranking - This is your ranking based on your Z Score for the analyte of interest among all possible rankings. It is important to note that several laboratories can achieve the same rank.

Z-Score - This figure corresponds to how far your Reported Value was from the EPA Mean, reported in terms of EPA Standard Deviation units. As a point of reference, the US EPA Acceptance Limits are plus or minus three (3) EPA Standard Deviations from the EPA Mean.

Laboratory Benchmark Report

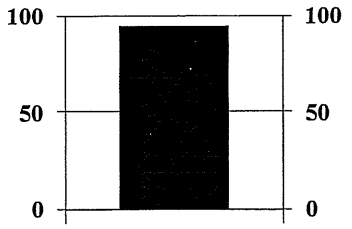
Customer Code: 3655 **WP October 2001**

Summary of Ranking Data

Summary of National Ranking Data

Total Number of Laboratories Reporting:	220
Average Percent Acceptable Data:	95.02%
Average Percentile for all Parameters:	51.19%

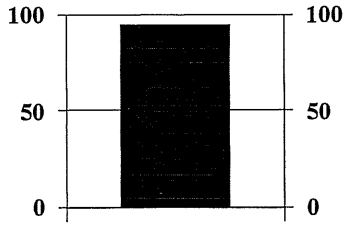
National Avg % Acceptable
95.0 %



Summary of Laboratory Ranking:

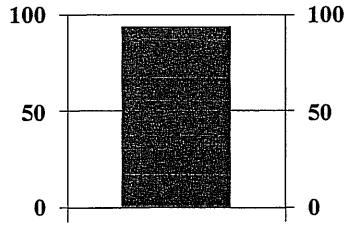
Total Data Points Reported:	195
Number of Acceptable Data Points:	184
Percent Acceptable Data:	94.36%
Average Percentile for all Parameters:	49.67%

Laboratory Avg % Acceptable
94.36%



Rank on Percent Acceptable Data:	28 of 61
Rank on Average Percentile Data:	104 of 197
Weighted Rank on Percent Acceptable Data:	13 of 94
Weighted Rank on Average Percentile Data:	13 of 209

Weighted Rank Avg Percentile
93.8 %



Comment Report

There are no comments to report.

Exception Report

WP October 2001
Study Closing Date 11/15/2001

Product: Pesticides **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
4,4'-DDD	WP	49	6.56	3.59	1.76-5.01	5.86	EPA608	Not Acceptable
4,4'-DDT	WP	51	8.10	4.76	2.18-6.44	5.33	EPA608	Not Acceptable
Aldrin	WP	47	2.73	1.51	0.384-2.04	5.51	EPA608	Not Acceptable
alpha-BHC	WP		11.6	6.89	2.53-9.55	4.75	EPA608	Not Acceptable
beta-BHC	WP		8.80	4.06	1.39-6	6.65	EPA608	Not Acceptable
Endosulfan I	WP		9.60	3.63	1.48-5.09	10.5	EPA608	Not Acceptable
Endosulfan II	WP		49.6	23.8	4.85-38	5.1	EPA608	Not Acceptable
Endosulfan sulfate	WP		18.0	8.31	2.08-13.2	5.61	EPA608	Not Acceptable
gamma-BHC	WP		10.4	4.61	1.63-6.6	7.6	EPA608	Not Acceptable
Heptachlor	WP	52	3.97	2.68	0.705-3.73	3.47	EPA608	Not Acceptable

Product: TPH Diesel (DRO) **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
TPH Diesel (DRO)	WP		2.81	1.03	0.369-1.29	13	OA-2	Not Acceptable

Performance Summary

WP October 2001
Study Closing Date 11/15/2001

Product: Acid Extractables **WP Lot Number: 31153** **APG+ Lot Number: 31185**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
2,4,5-Trichlorophenol	WP		47.1	57.7	26-72.8	0.295	EPA625	Acceptable
2,4,6-Trichlorophenol	WP		36.0	43.1	15.4-56.1	0.0295	EPA625	Acceptable
2,4-Dichlorophenol	WP		117	157	56.6-188	0.228	EPA625	Acceptable
2,4-Dimethylphenol	WP		61.1	79.9	15.9-106	0.0268	EPA625	Acceptable
2,4-Dinitrophenol	WP		<2.0				EPA625	Acceptable
2-Chlorophenol	WP		106	142	40.3-178	0.131	EPA625	Acceptable
2-Methyl-4,6-Dinitrophenol	WP		81.3	107	16.6-151	0.103	EPA625	Acceptable
2-Nitrophenol	WP		15.7	20.1	7-25.3	0.163	EPA625	Acceptable
4-Chloro-3-methylphenol	WP		10.5	12.8	3.16-16.6	0.267	EPA625	Acceptable
4-Nitrophenol	WP		55.7	88.8	0-123	0.251	EPA625	Acceptable
Pentachlorophenol	WP		99.8	114	29.9-158	0.266	EPA625	Acceptable
Phenol	WP		<2.0				EPA625	Acceptable

Product: Aluminum High Level **WP Lot Number: 31162** **APG+ Lot Number: 31179**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aluminum High Level	WP		0.338	0.3	0.231-0.375	1.45	EPA200.7	Acceptable

Performance Summary

Product: Base Neutral Extractables **WP Lot Number: 31154** **APG+ Lot Number: 31186**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
1,2,4-Trichlorobenzene	WP		24.2	34.1	9.62-43.6	0.424	EPA625	Acceptable
1,2-Dichlorobenzene	WP		35.3	51.5	21.7-56.8	0.683	EPA625	Acceptable
1,3-Dichlorobenzene	WP		<2.0				EPA625	Acceptable
1,4-Dichlorobenzene	WP		<2.0				EPA625	Acceptable
2,4-Dinitrotoluene (2,4-DNT)	WP		20.2	33.5	9.91-43.7	1.17	EPA625	Acceptable
2,6-Dinitrotoluene (2,6-DNT)	WP		47.9	62.9	24-81	0.484	EPA625	Acceptable
2-Chloronaphthalene	WP		81.9	103	41.4-118	0.172	EPA625	Acceptable
3,3'-Dichlorobenzidine	WP		<2.0				EPA625	Acceptable
4-Bromophenyl phenyl ether	WP		<2.0				EPA625	Acceptable
4-Chlorophenyl phenyl ether	WP		<2.0				EPA625	Acceptable
Acenaphthene	WP		46.9	52.9	20.5-67.6	0.357	EPA625	Acceptable
Acenaphthylene	WP		50.8	49.8	19.3-61.8	1.45	EPA625	Acceptable
Anthracene	WP		111	127	43.5-163	0.4	EPA625	Acceptable
Benzidine	WP		<2.0				EPA625	Acceptable
Benzo(a)anthracene	WP		68.4	75.9	39.5-92.8	0.248	EPA625	Acceptable
Benzo(a)pyrene	WP		13.4	15	0-20.9	0.934	EPA625	Acceptable
Benzo(b)fluoranthene	WP		<2.0				EPA625	Acceptable
Benzo(g,h,i)perylene	WP		<2.0				EPA625	Acceptable
Benzo(k)fluoranthene	WP		<2.0				EPA625	Acceptable
Bis(2-Chloroethoxy)methane	WP		<2.0				EPA625	Acceptable
Bis(2-Chloroethyl)ether	WP		<2.0				EPA625	Acceptable
Bis(2-Chloroisopropyl)ether	WP		<2.0				EPA625	Acceptable
Bis(2-Ethylhexyl)phthalate	WP		42.7	50.3	16.6-72.6	0.203	EPA625	Acceptable

Performance Summary

WP October 2001
Study Closing Date 11/15/2001

Product: Base Neutral Extractables **WP Lot Number: 31154** **APG+ Lot Number: 31186**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Butyl benzyl phthalate	WP		21.7	39.7	12.6-56.1	1.74	EPA625	Acceptable
Chrysene	WP		50.4	56.1	23.1-73.1	0.276	EPA625	Acceptable
Dibenzo(a,h)anthracene	WP		<2.0				EPA625	Acceptable
Diethyl phthalate	WP		81.0	111	0-169	0.0415	EPA625	Acceptable
Dimethyl phthalate	WP		49.1	119	0-183	0.825	EPA625	Acceptable
Di-n-butyl phthalate	WP		35.0	41.9	14-60.4	0.285	EPA625	Acceptable
Di-n-octyl phthalate	WP		82.8	98.3	15.1-143	0.168	EPA625	Acceptable
Fluoranthene	WP		25.9	29.6	15.2-37.7	0.16	EPA625	Acceptable
Fluorene	WP		109	127	53.1-162	0.0549	EPA625	Acceptable
Hexachlorobenzene	WP		20.3	22.5	9.75-29.5	0.213	EPA625	Acceptable
Hexachlorobutadiene	WP		25.9	41	5.47-51	0.303	EPA625	Acceptable
Hexachlorocyclopentadiene	WP		37.7	61.9	0-81.3	0.1	EPA625	Acceptable
Hexachloroethane	WP		29.4	48.8	6.4-60.3	0.434	EPA625	Acceptable
Indeno(1,2,3-cd)pyrene	WP		<2.0				EPA625	Acceptable
Isophorone	WP		43.8	40.6	16.5-52.8	1.52	EPA625	Acceptable
Naphthalene	WP		91.0	122	30.2-159	0.172	EPA625	Acceptable
Nitrobenzene (NB)	WP		79.0	102	33.4-133	0.259	EPA625	Acceptable
N-nitrosodimethylamine	WP		<2.0				EPA625	Acceptable
N-nitrosodi-n-propylamine	WP		<2.0				EPA625	Acceptable
N-nitrosodiphenylamine	WP		<2.0				EPA625	Acceptable
Phenanthrene	WP		64.9	70.7	29.9-91	0.431	EPA625	Acceptable
Pyrene	WP		54.8	60.9	20-85.1	0.202	EPA625	Acceptable

Performance Summary

Product: BTEX **WP Lot Number: 31156** **APG+ Lot Number: 31189**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Benzene	WP	65	14.1	13	9.01-17.2	0.735	EPA624	Acceptable
Ethylbenzene	WP	66	47.7	50.6	33.7-66.1	0.407	EPA624	Acceptable
m-Xylene / p-Xylene	WP		<2.0				EPA624	Acceptable
o-Xylene	WP		80.8	87.7	63.6-108	0.696	EPA624	Acceptable
Toluene	WP	67	9.95	9.11	6.18-12	0.901	EPA624	Acceptable
Total Xylenes	WP		80.8	87.7	49.8-119	0.313	EPA624	Acceptable

Product: Chlordane (Total) **WP Lot Number: 31150** **APG+ Lot Number: 31196**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Chlordane (Total)	WP	53	7.80	7.08	3.1-10.1	1.04	EPA608	Acceptable

Product: Demand **WP Lot Number: 31133** **APG+ Lot Number: 31167**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Biochemical Oxygen Demand	WP	38	54.8	55.6	27.9-83.2	0.0824	EPA405.1	Acceptable
Carbonaceous BOD	WP	102	46.9	47.9	21.4-74.3	0.109	SM5210B	Acceptable
Chemical Oxygen Demand	WP	36	82.8	89.7	65.8-106	0.449	EPA410.1	Acceptable
Total Organic Carbon	WP	37	35.6	35.4	29.5-41	0.157	EPA415.1	Acceptable

Performance Summary

Product: Fluoride **WP Lot Number: 31163** **APG+ Lot Number: 31180**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Fluoride	WP	29	2.14	2.39	2.08-2.68	2.42	EPA300.0	Check for Error

Product: Hexavalent Chromium **WP Lot Number: 31152** **APG+ Lot Number: 31182**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Chromium, Hexavalent	WP		79.0	77.9	60.2-88.4	0.998	EPA218.5	Acceptable

Product: Minerals **WP Lot Number: 31136-31137** **APG+ Lot Number: 31171-31172**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Alkalinity as CaCO3	WP	27	31.6	32.2	27.6-37.5	0.602	EPA310.1	Acceptable
Calcium	WP	23	21.5	20	17.6-23	1.35	EPA200.7	Acceptable
Chloride	WP	28	115	117	107-128	0.57	EPA300.0	Acceptable
Conductivity	WP	20	511	539	480-564	0.791	EPA120.1	Acceptable
Magnesium	WP	24	10.9	11.1	9.62-12.4	0.212	EPA200.7	Acceptable
Potassium	WP	26	32.0	31.5	27.2-35.9	0.276	EPA200.7	Acceptable
Sodium	WP	25	38.8	38.7	34.8-42.9	0.0735	EPA200.7	Acceptable
Sulfate	WP	30	20.8	21.9	17.1-26.1	0.537	EPA300.0	Acceptable
Total Dissolved Solids	WP	21	266	306	209-351	0.593	SM2510B	Acceptable
Total Hardness as CaCO3	WP	22	98.8	95.6	85.8-106	0.861	SM2340B	Acceptable

Performance Summary

Product: Nutrient **WP Lot Number: 31135-31441** **APG+ Lot Number: 31168-31169**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Ammonia Nitrogen as N	WP	31	10.5	10.5	8.11-12.7	0.13	EPA350.3	Acceptable
Nitrate Nitrogen as N	WP	32	19.0	18.5	14.7-22	0.574	EPA353.3	Acceptable
Orthophosphate as P	WP	33	3.41	3.79	3.23-4.38	2.09	EPA300.0	Check for Error
Total Kjeldahl Nitrogen	WP	34	8.88	11	7.84-13.8	1.94	EPA351.3	Acceptable
Total Phosphorus as P	WP	35		8.42	6.41-9.84			

Product: Oil & Grease **WP Lot Number: 31144** **APG+ Lot Number: 31170**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Oil & Grease	WP	104	36.1	38.1	25-43.7	0.577	EPA1664	Acceptable

Product: PCB's in Oil **WP Lot Number: 31147-31148**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aroclor 1016/1242 in Oil Sample 1	WP	99	<2.0				EPA608	Acceptable
Aroclor 1016/1242 in Oil Sample 2	WP	99	41.2	37.2	6.6-50.4	1.74	EPA608	Acceptable
Aroclor 1254 in Oil Sample 1	WP	100	18.5	18.4	0.62-29.8	0.679	EPA608	Acceptable
Aroclor 1254 in Oil Sample 2	WP	100	<2.0				EPA608	Acceptable
Aroclor 1260 in Oil Sample 1	WP	101	<2.0				EPA608	Acceptable
Aroclor 1260 in Oil Sample 2	WP	101	<2.0				EPA608	Acceptable

APG Customer 3655 Analytical Management Lab
EPA Lab Code KS00902 15130 South Keeler
Olathe, KS 66062

Performance Summary

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Product: PCB's in Water **WP Lot Number: 31145-31146** **APG+ Lot Number: 31193**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aroclor 1016/1242 Sample 1	WP	40	<0.2				EPA608	Acceptable
Aroclor 1016/1242 Sample 2	WP	40	12.0	12	3.11-17.1	0.815	EPA608	Acceptable
Aroclor 1221 Sample 1	WP		<0.2				EPA608	Acceptable
Aroclor 1221 Sample 2	WP		<0.2				EPA608	Acceptable
Aroclor 1232 Sample 1	WP	42	<0.2				EPA608	Acceptable
Aroclor 1232 Sample 2	WP	42	<0.2				EPA608	Acceptable
Aroclor 1248 Sample 1	WP	44	<0.2				EPA608	Acceptable
Aroclor 1248 Sample 2	WP	44	<0.2				EPA608	Acceptable
Aroclor 1254 Sample 1	WP	45	4.66	4.81	2.27-6.25	0.602	EPA608	Acceptable
Aroclor 1254 Sample 2	WP	45	<0.2				EPA608	Acceptable
Aroclor 1260 Sample 1	WP	46	<0.2				EPA608	Acceptable
Aroclor 1260 Sample 2	WP	46	<0.2				EPA608	Acceptable

Performance Summary

WP October 2001
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Product: Pesticides **WP Lot Number: 31149** **APG+ Lot Number: 31933**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
4,4'-DDD	WP	49	6.56	3.59	1.76-5.01	5.86	EPA608	**Not Acceptable**
4,4'-DDE	WP	50	1.77	2.33	1.1-3.17	1.07	EPA608	Acceptable
4,4'-DDT	WP	51	8.10	4.76	2.18-6.44	5.33	EPA608	**Not Acceptable**
Aldrin	WP	47	2.73	1.51	0.384-2.04	5.51	EPA608	**Not Acceptable**
alpha-BHC	WP		11.6	6.89	2.53-9.55	4.75	EPA608	**Not Acceptable**
beta-BHC	WP		8.80	4.06	1.39-6	6.65	EPA608	**Not Acceptable**
delta-BHC	WP		9.13	8.44	2.67-10.6	1.86	EPA608	Acceptable
Dieldrin	WP	48	3.26	3.06	1.58-4.23	0.792	EPA608	Acceptable
Endosulfan I	WP		9.60	3.63	1.48-5.09	10.5	EPA608	**Not Acceptable**
Endosulfan II	WP		49.6	23.8	4.85-38	5.1	EPA608	**Not Acceptable**
Endosulfan sulfate	WP		18.0	8.31	2.08-13.2	5.61	EPA608	**Not Acceptable**
Endrin	WP		<0.1				EPA608	Acceptable
Endrin aldehyde	WP		<0.1				EPA608	Acceptable
gamma-BHC	WP		10.4	4.61	1.63-6.6	7.6	EPA608	**Not Acceptable**
Heptachlor	WP	52	3.97	2.68	0.705-3.73	3.47	EPA608	**Not Acceptable**
Heptachlor epoxide (beta)	WP	78	2.75	2.15	1.09-2.76	2.95	EPA608	**Check for Error**
Methoxychlor	WP		5.75	4.19	1.43-6.68	1.93	EPA608	Acceptable

Product: pH **WP Lot Number: 31138** **APG+ Lot Number: 31173**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
pH	WP	19	7.47	7.48	7.27-7.68	0.147	EPA150.1	Acceptable

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Olathe, KS 66062

Performance Summary

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Product: Polynuclear Aromatic Hydrocarbons-PAH **WP Lot Number: 31155** **APG+ Lot Number: 31188**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Acenaphthene	WP		11.7	16	9.29-19.4	1.6	EPA625	Acceptable
Acenaphthylene	WP		<0.2				EPA625	Acceptable
Anthracene	WP		1.88	1.72	1.14-2.26	0.963	EPA625	Acceptable
Benzo(a)anthracene	WP		<0.2				EPA625	Acceptable
Benzo(a)pyrene	WP		<0.2				EPA625	Acceptable
Benzo(b)fluoranthene	WP		<0.2				EPA625	Acceptable
Benzo(g,h,i)perylene	WP		<0.2				EPA625	Acceptable
Benzo(k)fluoranthene	WP		<0.2				EPA625	Acceptable
Chrysene	WP		<0.2				EPA625	Acceptable
Dibenzo(a,h)anthracene	WP		<0.2				EPA625	Acceptable
Fluoranthene	WP		1.78	1.77	1.19-2.14	0.759	EPA625	Acceptable
Fluorene	WP		2.90	4.28	2.18-5.45	1.69	EPA625	Acceptable
Indeno(1,2,3-cd)pyrene	WP		<0.2				EPA625	Acceptable
Naphthalene	WP		13.4	19.9	10.3-23.3	1.56	EPA625	Acceptable
Phenanthrene	WP		<0.2				EPA625	Acceptable
Pyrene	WP		0.581	0.512	0.455-0.658	0.74	EPA625	Acceptable

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EPA Lab Code KS00902 15130 South Keeler
Olathe, KS 66062

Performance Summary

WP October 2001
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Product: Solids **WP Lot Number: 75071**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Dissolved Solids	WP		399	373	229-541	0.269	EPA160.1	Acceptable
Total Solids	WP		440	417	325-513	0.671	EPA160.3	Acceptable
Total Suspended Solids	WP	72	39	43.9	32.8-47	0.381	EPA160.2	Acceptable

Product: Total Cyanide **WP Lot Number: 31142** **APG+ Lot Number: 31177**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Cyanide	WP	71	0.547	0.681	0.472-0.876	1.89	EPA335.2	Acceptable

Product: Total Phenolics **WP Lot Number: 31141** **APG+ Lot Number: 31176**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Phenolics	WP	97		2.45	0.896-2.35			

Product: Total Residual Chlorine **WP Lot Number: 31143** **APG+ Lot Number: 31178**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Total Residual Chlorine	WP	98		2.27	1.8-2.64			

Product: Toxaphene **WP Lot Number: 31159** **APG+ Lot Number: 31194**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Toxaphene	WP		7.16	9.39	3.81-14.9	1.18	EPA608	Acceptable

Performance Summary

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Product: TPH 418.1 **WP Lot Number: 31166** **APG+ Lot Number: 31192**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
TPH by 418.1	WP			45.7	51.1-54			

Product: TPH Diesel (DRO) **WP Lot Number: 31157** **APG+ Lot Number: 31191**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
TPH Diesel (DRO)	WP		2.81	1.03	0.369-1.29	13	OA-2	**Not Acceptable**

Product: TPH Gasoline (GRO) **WP Lot Number: 31158** **APG+ Lot Number: 31190**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
TPH Gasoline (GRO)	WP		1.43	2.61	0.778-3.46	1.54	OA-1	Acceptable

Performance Summary

WP October 2001
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Product: Trace Metals **WP Lot Number: 31139-31140** **APG+ Lot Number: 31174-31175**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Aluminum	WP	1	1609	1670	1430-1900	0.659	EPA200.8	Acceptable
Antimony	WP	16	150	148	93.7-182	0.816	EPA200.8	Acceptable
Arsenic	WP	2	263	242	201-286	1.41	EPA200.8	Acceptable
Barium	WP		1204	1220	1040-1380	0.105	EPA200.7	Acceptable
Beryllium	WP	3	187	181	153-204	1.05	EPA200.8	Acceptable
Boron	WP			420	367-518			
Cadmium	WP	4	76.2	75.7	63.8-87.2	0.179	EPA200.8	Acceptable
Chromium	WP	6	221	223	192-253	0.196	EPA200.8	Acceptable
Cobalt	WP	5	192	180	157-203	1.58	EPA200.7	Acceptable
Copper	WP	7	523	520	471-571	0.12	EPA200.7	Acceptable
Iron	WP	8	2972	3000	2660-3370	0.403	EPA200.8	Acceptable
Lead	WP	12	1047	1070	942-1200	0.535	EPA200.8	Acceptable
Manganese	WP	10	1226	1200	1080-1330	0.376	EPA200.8	Acceptable
Mercury	WP	9	6.15	6.53	4.82-8.22	0.654	EPA245.2	Acceptable
Molybdenum	WP	74	472	437	375-500	1.68	EPA200.7	Acceptable
Nickel	WP	11	1300	1210	1100-1350	1.89	EPA200.8	Acceptable
Selenium	WP	13	577	582	461-674	0.281	EPA200.7	Acceptable
Silver	WP	17	275	251	215-288	1.9	EPA200.7	Acceptable
Strontium	WP	75		209	177-239			
Thallium	WP	18	249	257	206-301	0.253	EPA200.8	Acceptable
Titanium	WP	76		72.6	61.3-83			
Vanadium	WP	14	1217	1200	1080-1320	0.431	EPA200.7	Acceptable
Zinc	WP	15	1558	1500	1330-1680	0.822	EPA200.8	Acceptable

Performance Summary

Product: Volatiles **WP Lot Number: 31151** **APG+ Lot Number: 31184**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
1,1,1-Trichloroethane	WP	56	58.2	58.6	37.8-77.3	0.091	EPA624	Acceptable
1,1,2,2-Tetrachloroethane	WP		<2.0				EPA624	Acceptable
1,1,2-Trichloroethane	WP		48.8	51.7	35.5-67	0.475	EPA624	Acceptable
1,1-Dichloroethane	WP		<2.0				EPA624	Acceptable
1,1-Dichloroethene	WP		159	163	92.6-245	0.394	EPA624	Acceptable
1,2-Dichlorobenzene	WP	94	22.1	21.4	14.8-27.5	0.474	EPA624	Acceptable
1,2-Dichloroethane	WP	54	62.0	69.1	47.6-91.7	1.04	EPA624	Acceptable
1,2-Dichloropropane	WP		<2.0				EPA624	Acceptable
1,3-Dichlorobenzene	WP	96	19.1	18.4	12.8-23.3	0.568	EPA624	Acceptable
1,4-Dichlorobenzene	WP	95	55.5	60.4	41.6-77.7	0.698	EPA624	Acceptable
Benzene	WP	65	33.3	29.4	21-38.1	1.3	EPA624	Acceptable
Bromodichloromethane	WP	60	26.8	23.2	16.3-30.3	1.49	EPA624	Acceptable
Bromoform	WP	62	39.8	46.7	30.3-64	1.32	EPA624	Acceptable
Bromomethane	WP		<2.0				EPA624	Acceptable
Carbon tetrachloride	WP	58	27.1	24.5	15.3-34.6	0.654	EPA624	Acceptable
Chlorobenzene	WP	64	15.9	14.9	11.1-18.8	0.698	EPA624	Acceptable
Chloroethane	WP		<2.0				EPA624	Acceptable
Chloroform	WP	55	37.6	39.6	27.6-50.8	0.412	EPA624	Acceptable
Chloromethane	WP		<2.0				EPA624	Acceptable
cis-1,3-Dichloropropene	WP		<2.0				EPA624	Acceptable
Dibromochloromethane	WP	61	21.8	19.3	12.7-25.6	1.21	EPA624	Acceptable
Ethylbenzene	WP	66	37.4	40.9	27.3-53.4	0.668	EPA624	Acceptable
Methylene chloride	WP	63	17.3	21.3	13.5-29.9	1.61	EPA624	Acceptable

Performance Summary

WP October 2001
Study Closing Date 11/15/2001

Product: Volatiles **WP Lot Number: 31151** **APG+ Lot Number: 31184**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Test Method	Evaluation
Tetrachloroethene	WP	59	37.1	41.8	27.6-53.6	0.806	EPA624	Acceptable
Toluene	WP	67	33.3	29.5	21.1-37.2	1.56	EPA624	Acceptable
trans-1,2-Dichloroethene	WP		<2.0				EPA624	Acceptable
trans-1,3-Dichloropropene	WP		<2.0				EPA624	Acceptable
Trichloroethene	WP	57	63.0	68.7	44.5-88.6	0.489	EPA624	Acceptable
Trichlorofluoromethane	WP		<2.0				EPA624	Acceptable
Vinyl Chloride	WP		<2.0				EPA624	Acceptable

Analytical Products Group, Inc.

PERFORMANCE REPORT

WP Performance Summary

April 2002

APG Customer Code: 3655

Analytical Management Lab
Suite B
15130 South Keeler
Olathe, KS 66062

Laboratory of
EXCELLENCE

NVLAP®
LAB CODE 200384-0

Water Pollution (WP) Proficiency Testing Program Report

Introduction:

The Analytical Products Group, Inc. Proficiency Testing Program has been designed to evaluate your laboratory's performance against other participating laboratories on the same set of standards. This report has been specifically prepared for your organization to assist you in ensuring that your procedures are in-control. Therefore, in most cases sample levels are not designed to challenge the detection limits of the method.

The Analytical Products Group Proficiency Testing Program is certified to meet all of the following requirements:

- Accredited by NIST according to the National Voluntary Laboratory Accreditation Program (Handbook 150-19) for Chemical Calibrations: Providers of Proficiency Testing (NVLAB Code 200384-0)
- Analytical Products Group, Inc. Quality System is registered with DNVCertification to meet the ISO 9001 standard to design, manufacture, and distribute chemical reference materials and operate Proficiency Testing Programs (Certificate # 98-HOU-AQ-9513)
- Proficiency Testing Program is certified to meet all requirements as specified in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).
- Certified to meet ISO/REMCO Harmonized Protocol for Proficiency Testing of Analytical Laboratories
- Certified to meet ASTM E1301

Report Layout

This report contains the following sub-reports:

Introduction -This section is the general introduction to the Analytical Products Group, Inc. Proficiency Testing Program as well as all of the certifications that the organization maintains.

Terms & Definitions - Defines all of the terms used throughout the report. This is an excellent section to refer to first if you have any questions.

Laboratory Benchmark Report - This report presents an overall summary of the study as well as your performance relative to all other study participants.

Comment Report - Contains comments submitted to Analytical Products Group by the program participants or comments by Analytical Products Group to program participants concerning unusual characteristics of the data set or problems with standards which could influence the interpretation of the data. Please review this section thoroughly before examining your data.

Exception Report - This report is a summary of all of the analytes that resulted in an Evaluation of "Not Acceptable." The Exception Report is designed to very quickly identify for your laboratory the areas that may require your immediate attention.

Performance Evaluation Summary Report - Provides a summary of all of your data for each analyte. This summary is formatted similar to the US EPA DMR-QA Reports.

Detail Report- This report lists all the details of your data. Also included in this section are useful graphs indicating the APG Assigned Value, the EPA Mean, the EPA Acceptance Limits and your Reported Value. Another graph included in this section shows your Z score for the analyte of interest.

The goal of this report is to provide your laboratory with the information you need to monitor and improve your quality. If you have any questions or feedback, please give us a call (800-272-4442). Thank you for choosing Analytical Products Group as your proficiency testing provider.

Sincerely,

Thomas J. Coyner

Thomas J. Coyner
Proficiency Program Manager

Terms & Definitions

Average Acceptable Data - This value is the average percent of data that is within the EPA Acceptance Limits for this study. The Average Acceptable Data is reported for all laboratories as a whole as well as your laboratory as an individual. These values can be found in the Laboratory Benchmark Report.

Average Percentile for All Parameters - This is your laboratory's rank on each parameter and the related rank for that parameter. The average percentile is the average of this data across all parameters reported. It is a different measure of performance than percent acceptable data since it is affected by your relative performance on each parameter. The report provides an average for this measure for all laboratories reporting as well as your laboratory's performance.

Analyte - Name of the analyte of interest as found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) or NELAC Field of Testing List.

Analyte Code - This value is the code given to the analyte by the US EPA. These codes can be found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045). If no code is listed, then this analyte is not part of the US EPA program.

APG Assigned Value - This value is the calculated True Value of the standard based upon the actual composition of the standard.

APG Certified Value - This value is the result of the quality control testing performed on the sample by Analytical Products Group before the sample is shipped to the customer. Through this testing, the samples are traced against a NIST Standard Reference Material where available or an external standard if a NIST Standard Reference Material is not available. The reported uncertainty of this value is expressed as two times the standard deviation (s) divided by the square root of the number of samples (n) tested (i.e. 2s/vn). This quality control testing meets all of the requirements as specified by the NIST National Voluntary Laboratory Accreditation Program (NVLAP) Handbook 150-19 (Chemical Calibration: Providers of Proficiency Testing).

EPA Acceptance Range - This range is calculated based upon the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045). For the Water Pollution Program (WP), EPA Acceptance Limits are defined as ± three (3) EPA Standard Deviations from the EPA Mean.

EPA Mean - If the analyte is included in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then this value is the Mean Value calculated per the Criteria Document. If the analyte is not included in the Criteria Document, then this value is the Study Mean.

Terms & Definitions (continued)

Evaluation - This value is determined by the EPA Acceptance Limits and the Reported Value. Evaluation may have one of following values:

Value	Description
Acceptable	The Reported Value was within the EPA Acceptable Limits or a less than (<) value was reported AND zero (0) was included in the EPA Acceptance Limit Range AND the numeric part of the Reported Value was less than the upper limit of the EPA Acceptance Limit.
Not Acceptable	The Reported Value is outside of the EPA Acceptance Limits or a less than (<) value was reported AND zero (0) is not included in the EPA Acceptance Limits range or the numeric part of the Reported Value was greater than the upper limit of the EPA Acceptance Limit or a greater than value (>) was reported.
Check for Error	The Reported Value is between two (2) and three (3) EPA Standard Deviations from the EPA Mean.
Unusable	The Reported Value was a text value which could not be evaluated.
Blank	No data was reported for this analyte or the analyte was not spiked.

Inter-Laboratory Statistics - This data is a summary of all of the data from all study participants for the analyte of interest. Inter-Laboratory Statistics are only reported if a minimum number of laboratories analyzed and reported data for the analyte of interest.

Laboratory Pass Percentage - This is the percentage of study participants that reported values within the EPA Acceptance Limits for the analyte of interest.

Number of Acceptable Data Points - This value is the number of data points reported by your laboratory that were within the EPA Acceptance Limits.

Program - This value indicates which program the analyte of interest is a part. For Water Pollution Studies (WP), the only values that Program will have are 'WP' or 'APG+'. If the analyte and range are consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then the Program value will have a value of 'WP' and if this is not the case then the Program value will be 'APG+', indicating that the analyte and/or range was specified by Analytical Products Group.

Rank on Percent Acceptable Data - This is your laboratories rank out of all possible ranks in the current study based on percent acceptable data. It is important to note that several laboratories can achieve the same rank.

Rank on Average Percentile Data - This value is your laboratories rank out of all possible ranks based on Your Ranking converted to a percentile. It is important to note that several laboratories can achieve the same rank.

Reported Value - This value is the result that was reported by your organization for the particular analyte. Every effort has been made to ensure correct data entry. Should you find a data entry error, please notify Analytical Products Group, Inc. and we will correct the error and re-issue the report. If the Reported Value is reported as a less than (<) than figure, then the only way that the Evaluation will result in an "Acceptable" value is if the numeric part of the Reported Value (i.e. 8 if the Report Value were <8) is less then the upper limit of the EPA Acceptance Limit AND zero (0) is included in the EPA Acceptance Limit. Any other non-numeric value (i.e. BDL, ND, >10, etc.) cannot be evaluated and will return an Evaluation result of "Unusable." This method of evaluation is consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).

Solids Standards - Solids standards are individually prepared, therefore, each standard has a unique APG Assigned Value.

Study Mean - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Mean is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Terms & Definitions (continued)

Study Standard Deviation - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Standard Deviation is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Weighted Rank on Percent Acceptable Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percent Acceptable Data is the weighted rank of your laboratory based on Percent Acceptable Data and the number of analytes reported.

Weight Rank on Percentile Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percentile Data is the weighted rank of your laboratory based on Rank on Average Percentile Data and the number of analytes reported.

Your Ranking - This is your ranking based on your Z Score for the analyte of interest among all possible rankings. It is important to note that several laboratories can achieve the same rank.

Z-Score - This figure corresponds to how far your Reported Value was from the EPA Mean, reported in terms of EPA Standard Deviation units. As a point of reference, the US EPA Acceptance Limits are plus or minus three (3) EPA Standard Deviations from the EPA Mean.

Laboratory Benchmark Report

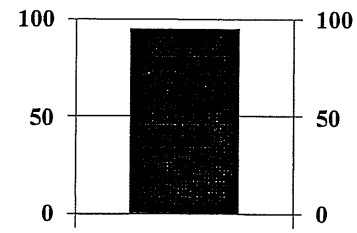
Customer Code: 3655 **WP April 2002**

Summary of Ranking Data

Summary of National Ranking Data

Total Number of Laboratories Reporting:	189
Average Percent Acceptable Data:	94.68%
Average Percentile for all Parameters:	51.34%

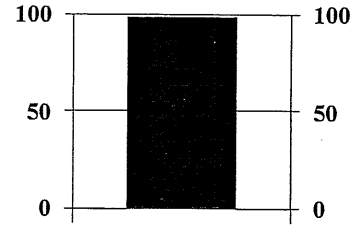
National Avg % Acceptable
94.7 %



Summary of Laboratory Ranking:

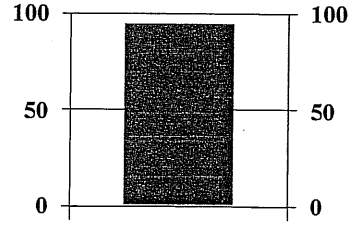
Total Data Points Reported:	204
Number of Acceptable Data Points:	200
Percent Acceptable Data:	98.04%
Average Percentile for all Parameters:	47.87%

Laboratory Avg % Acceptable
98.04%



Rank on Percent Acceptable Data:	12 of 65
Rank on Average Percentile Data:	122 of 176
Weighted Rank on Percent Acceptable Data:	9 of 95
Weighted Rank on Average Percentile Data:	12 of 186

Weighted Rank Avg Percentile
93.5 %



Comments

Volatiles:

One third of the laboratories reporting Bromomethane also reported Chloromethane, which was not spiked into the sample. APG has reviewed the original pre-study quality control data, rerun the sample after the study close and has verified that the Chloromethane is not present. However, nine of the ten reporting laboratories are within a limited geography. APG is evaluating whether or not all laboratories received the sample through a single shipping point that might have mishandled the shipment. We are also evaluating the effect of high temperature storage on the sample. Laboratories involved will be notified of the results.

Exception Report

WP April 2002
Study Closing Date 05/15/2002

Product: Demand **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Organic Carbon	WP	2040	99.3	72.5	60.6-83.4	7.2		EPA 415.1	Not Acceptable

Product: Herbicides **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Dicamba	WP	8595	0.36	5.44	0.409-7.97	3.04	10105609	EPA 615	Not Acceptable

Product: Pesticides (WP) **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
4,4'-DDD	WP	7355	1.43	0.822	0.391-1.16	5.12		EPA 608	Not Acceptable

Product: Trace Metals **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Manganese	WP	1090	1675	1500	1350-1670	3.11	10014401	EPA 200.8	Not Acceptable

Performance Summary

Product: Acid Extractables **WP Lot Number: 32713** **APG+ Lot Number: 32748**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
2,4,5-Trichlorophenol	WP	6835	74.0	80.5	33-102	0.565	10107401	EPA 625	Acceptable
2,4,6-Trichlorophenol	WP	6840	<2.0				10107401	EPA 625	Acceptable
2,4-Dichlorophenol	WP	6000	35.7	39.7	14.4-50.2	0.571	10107401	EPA 625	Acceptable
2,4-Dimethylphenol	WP	6130	53.8	60.9	11.2-81	0.664	10107401	EPA 625	Acceptable
2,4-Dinitrophenol	WP	6175	<2.0				10107401	EPA 625	Acceptable
2-Chlorophenol	WP	5800	53.8	59.4	18.5-75.5	0.716	10107401	EPA 625	Acceptable
2-Methyl-4,6-Dinitrophenol	WP	6360	137	180	17-265	0.0966	10107401	EPA 625	Acceptable
2-Nitrophenol	WP	6490	74.3	83.8	25.3-110	0.475	10107401	EPA 625	Acceptable
4-Chloro-3-methylphenol	WP	5700	46.7	50	18.3-64.4	0.69	10107401	EPA 625	Acceptable
4-Nitrophenol	WP	6500	<2.0	75.4	0-106		10107401	EPA 625	Acceptable
Pentachlorophenol	WP	6605	78.7	81.3	19.4-112	0.826	10107401	EPA 625	Acceptable
Phenol	WP	6625	112	176	0-235	0.294	10107401	EPA 625	Acceptable

Product: Aluminum High Level **WP Lot Number: 32722** **APG+ Lot Number: 32742**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Aluminum High Level	WP		0.934	0.901	0.728-1.01	1.32		EPA 200.7	Acceptable

Performance Summary

WP April 2002
Study Closing Date 05/15/2002

Product: Aromatic Hydrocarbons **WP Lot Number: 32727** **APG+ Lot Number: 32762**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
1,2-Dichlorobenzene	WP	4610	44.4	41.8	29.4-53	0.816	10107207	EPA 624	Acceptable
1,3-Dichlorobenzene	WP	4615	30.9	30.3	21.3-38	0.466	10107207	EPA 624	Acceptable
1,4-Dichlorobenzene	WP	4620	9.13	8.16	5.43-11.1	0.933	10107207	EPA 624	Acceptable
Benzene	WP	4375	20.8	19.6	13.8-25.6	0.561	10107207	EPA 624	Acceptable
Ethylbenzene	WP	4765	10.8	10.3	7.17-13.2	0.594	10107207	EPA 624	Acceptable
m-Xylene / p-Xylene	WP	5240	98.4	100	75.2-122	0.0509	10107207	EPA 624	Acceptable
o-Xylene	WP	5250	102	99.3	73.3-127	0.222	10107207	EPA 624	Acceptable
Toluene	WP	5140	64.4	60.5	43.6-75.4	0.925	10107207	EPA 624	Acceptable
Total Xylenes	WP	5260	200	200	115-267	0.354	10107207	EPA 624	Acceptable

Performance Summary

WP April 2002
Study Closing Date 05/15/2002

Product: Base Neutral Extractables **WP Lot Number: 32714** **APG+ Lot Number: 32749**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
1,2,4-Trichlorobenzene	WP	5155	111	168	49.4-197	0.486	10107401	EPA 625	Acceptable
1,2-Dichlorobenzene	WP	4610	34.9	45.1	17.7-50.4	0.165	10107401	EPA 625	Acceptable
1,3-Dichlorobenzene	WP	4615	63.9	84.9	23.1-100	0.18	10107401	EPA 625	Acceptable
1,4-Dichlorobenzene	WP	4620	<2.0				10107401	EPA 625	Acceptable
2,4-Dinitrotoluene (2,4-DNT)	WP	6185	121	144	61.1-192	0.275	10107401	EPA 625	Acceptable
2,6-Dinitrotoluene (2,6-DNT)	WP	6190	47.3	49.5	18.9-62.9	0.873	10107401	EPA 625	Acceptable
2-Chloronaphthalene	WP	5795	108	151	60.9-170	0.387	10107401	EPA 625	Acceptable
3,3'-Dichlorobenzidine	WP	5945	<2.0				10107401	EPA 625	Acceptable
4-Bromophenyl phenyl ether	WP	5660	<2.0				10107401	EPA 625	Acceptable
4-Chlorophenyl phenyl ether	WP	5825	<2.0				10107401	EPA 625	Acceptable
Acenaphthene	WP	5500	35.2	40.2	16-51.4	0.254	10107401	EPA 625	Acceptable
Acenaphthylene	WP	5505	47.8	42.1	16.1-52.6	2.22	10107401	EPA 625	Check for Error
Anthracene	WP	5555	68.3	75.3	28.6-97.3	0.47	10107401	EPA 625	Acceptable
Benzidine	WP	5595	<2.0				10107401	EPA 625	Acceptable
Benzo(a)anthracene	WP	5575	54.7	56.5	30.4-68	0.877	10107401	EPA 625	Acceptable
Benzo(a)pyrene	WP	5580	115	121	33.8-156	0.975	10107401	EPA 625	Acceptable
Benzo(b)fluoranthene	WP	5585	<2.0				10107401	EPA 625	Acceptable
Benzo(g,h,i)perylene	WP	5590	<2.0				10107401	EPA 625	Acceptable
Benzo(k)fluoranthene	WP	5600	<2.0				10107401	EPA 625	Acceptable
Bis(2-Chloroethoxy)methane	WP	5760	<2.0				10107401	EPA 625	Acceptable
Bis(2-Chloroethyl)ether	WP	5765	<2.0				10107401	EPA 625	Acceptable
Bis(2-Chloroisopropyl)ether	WP	5780	<2.0				10107401	EPA 625	Acceptable
Bis(2-Ethylhexyl)phthalate	WP	6255	146	179	22.1-263	0.0998	10107401	EPA 625	Acceptable

APG Customer 3655 Analytical Management Lab
EPA Lab Code KS00902 15130 South Keeler
Olathe, KS 66062

Performance Summary

WP April 2002
Study Closing Date 05/15/2002

Product: Base Neutral Extractables **WP Lot Number: 32714** **APG+ Lot Number: 32749**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Butyl benzyl phthalate	WP	5670	162	190	0-283	0.611	10107401	EPA 625	Acceptable
Chrysene	WP	5855	39.0	42.7	18.6-55.2	0.344	10107401	EPA 625	Acceptable
Dibenzo(a,h)anthracene	WP	5895	<2.0				10107401	EPA 625	Acceptable
Diethyl phthalate	WP	6070	86.4	85.4	0.796-130	0.968	10107401	EPA 625	Acceptable
Dimethyl phthalate	WP	6135	98.1	102	0-156	1.01	10107401	EPA 625	Acceptable
Di-n-butyl phthalate	WP	5925	118	112	15.6-157	1.34	10107401	EPA 625	Acceptable
Di-n-octyl phthalate	WP	6200	78.5	74.5	12.9-109	1.07	10107401	EPA 625	Acceptable
Fluoranthene	WP	6265	127	129	54.8-167	0.856	10107401	EPA 625	Acceptable
Fluorene	WP	6270	120	129	54-165	0.595	10107401	EPA 625	Acceptable
Hexachlorobenzene	WP	6275	118	155	67.9-200	0.727	10107401	EPA 625	Acceptable
Hexachlorobutadiene	WP	4835	116	178	31.8-212	0.199	10107401	EPA 625	Acceptable
Hexachlorocyclopentadiene	WP	6285	12.1	13.9	0-21.9	0.808	10107401	EPA 625	Acceptable
Hexachloroethane	WP	4840	58.1	76.5	7.92-96.1	0.415	10107401	EPA 625	Acceptable
Indeno(1,2,3-cd)pyrene	WP	6315	<2.0				10107401	EPA 625	Acceptable
Isophorone	WP	6320	69.1	56.8	21.5-74.7	2.37	10107401	EPA 625	Check for Error
Naphthalene	WP	5005	43.9	54.8	16.1-71.9	0.0107	10107401	EPA 625	Acceptable
Nitrobenzene (NB)	WP	5015	130	190	60.1-249	0.764	10107401	EPA 625	Acceptable
N-nitrosodimethylamine	WP	6530	<2.0				10107401	EPA 625	Acceptable
N-nitrosodi-n-propylamine	WP	6545	<2.0				10107401	EPA 625	Acceptable
N-nitrosodiphenylamine	WP	6535	<2.0				10107401	EPA 625	Acceptable
Phenanthrene	WP	6615	<2.0				10107401	EPA 625	Acceptable
Pyrene	WP	6665	34.0	37.8	12.2-55.1	0.042	10107401	EPA 625	Acceptable

Performance Summary

Product: BTEX **WP Lot Number: 32716** **APG+ Lot Number: 32752**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Benzene	WP	4375	13.7	12.8	8.87-16.9	0.597	10107207	EPA 624	Acceptable
Ethylbenzene	WP	4765	9.62	10.1	7.04-12.9	0.376	10107207	EPA 624	Acceptable
m-Xylene / p-Xylene	WP	5240	17.8	20.5	15.9-24.4	1.63	10107207	EPA 624	Acceptable
o-Xylene	WP	5250	<2.0				10107207	EPA 624	Acceptable
Toluene	WP	5140	31.5	31.2	22.3-39.2	0.283	10107207	EPA 624	Acceptable
Total Xylenes	WP	5260	17.8	20.5	10.9-30.4	0.859	10107207	EPA 624	Acceptable

Product: Chlordane (Total) **WP Lot Number: 32710** **APG+ Lot Number: 32759**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Chlordane (Total)	WP	7250	2.18	2.01	0.884-2.85	0.945	10103603	EPA 608	Acceptable

Product: Demand **WP Lot Number: 32693** **APG+ Lot Number: 32730**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Biochemical Oxygen Demand	WP	1530	72.3	113	57.2-169	2.19		EPA 405.1	Check for Error
Carbonaceous BOD	WP	1555	63.6	97.6	43.8-151	1.9		EPA 405.1	Acceptable
Chemical Oxygen Demand	WP	1565	185	183	141-208	0.901	10075806	EPA 410.1	Acceptable
Total Organic Carbon	WP	2040	99.3	72.5	60.6-83.4	7.2		EPA 415.1	**Not Acceptable**

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Product: Fluoride **WP Lot Number: 32723** **APG+ Lot Number: 32743**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Fluoride	WP	1730	0.267	0.239	0.176-0.304	1.27		EPA 300.0	Acceptable

Product: Herbicides **WP Lot Number: 32725** **APG+ Lot Number: 32760**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
2,4,5-T	WP	8655	0.43	3.48	0.0955-5.35	2.62	10105609	EPA 615	Check for Error
2,4,5-T	APG+			7.93	0.847-11.6				
2,4,5-TP (silvex)	WP	8650	1.55	6.39	0.322-8.62	2.12	10105609	EPA 615	Check for Error
2,4,5-TP (silvex)	APG+			17.8	1.17-22.9				
2,4-D	WP	8545	1.20	9.56	0-14.3	2.38	10105609	EPA 615	Check for Error
2,4-D	APG+			17.7	0-25.5				
Dicamba	WP	8595	0.36	5.44	0.409-7.97	3.04	10105609	EPA 615	**Not Acceptable**
Dicamba	APG+			7.37	0.635-10.7				
Dinoseb	WP	8620	11.9	7.15	0-15.7	1.99	10105609	EPA 615	Acceptable
Dinoseb	APG+			14.1	0-17				

Product: Hexavalent Chromium **WP Lot Number: 32712** **APG+ Lot Number: 32745**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Chromium, Hexavalent	WP	1045	80	77.9	64-93.4	0.265	10162206	EPA 7196	Acceptable

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Product: Minerals **WP Lot Number: 32696-32697** **APG+ Lot Number: 32734-32735**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Alkalinity as CaCO3	WP	1505	49.3	47.6	42-53.7	0.773	20003003	SM 18/19thED 23	Acceptable
Calcium	WP	1035	6.45	6.4	5.37-7.61	0.107	10013602	EPA 200.7	Acceptable
Chloride	WP	1575	48.3	51.5	45.9-56.6	1.68	10053006	EPA 300.0	Acceptable
Conductivity	WP	1610	312	317	292-340	0.503	20003809	SM 18/19thED 25	Acceptable
Magnesium	WP	1085	7.68	7.7	6.67-8.7	0.0297	10013602	EPA 200.7	Acceptable
Potassium	WP	1125	11.3	12.8	10.9-14.8	2.3	10013602	EPA 200.7	Check for Error
Sodium	WP	1155	34.9	35	31.4-38.9	0.24	10013602	EPA 200.7	Acceptable
Sulfate	WP	2000	19.4	19.2	14.9-23.1	0.292	10053006	EPA 300.0	Acceptable
Total Dissolved Solids	WP	1705		169	111-201				
Total Hardness as CaCO3	WP	1755	47.9	47.7	41.5-54.4	0.0465		SM 2340B	Acceptable

Product: Nutrient **WP Lot Number: 32694-32695** **APG+ Lot Number: 32731-32732**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Ammonia Nitrogen as N	WP	1515	5.13	4.7	3.59-5.78	1.23	10064207	EPA 350.3	Acceptable
Nitrate Nitrogen as N	WP	1810	15.4	16.7	13.2-19.8	1		EPA 300.0	Acceptable
Orthophosphate as P	WP	1870	5.46	5.41	4.62-6.24	0.111		EPA 300.0	Acceptable
Total Kjeldahl Nitrogen	WP	1795		7.85	5.52-10				
Total Phosphorus as P	WP	1910		4.34	3.3-5.09				

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WP April 2002
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Product: Oil & Grease **WP Lot Number: 32704** **APG+ Lot Number: 32733**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Oil & Grease	WP	1860	4.9	9.02	3.39-13.4	2.1		EPA 1664	Check for Error

Product: PCB's in Oil **WP Lot Number: 32707-32708**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Aroclor 1016/1242 in Oil Sample 1	WP		<1.7					EPA 608	Acceptable
Aroclor 1016/1242 in Oil Sample 2	WP		<1.7					EPA 608	Acceptable
Aroclor 1254 in Oil Sample 1	WP	8905	<1.7					EPA 608	Acceptable
Aroclor 1254 in Oil Sample 2	WP	8905	15.9	16.6	0.131-27.3	0.486		EPA 608	Acceptable
Aroclor 1260 in Oil Sample 1	WP	8910	51.3	48.7	8.79-68.4	1.28		EPA 608	Acceptable
Aroclor 1260 in Oil Sample 2	WP	8910	<1.7					EPA 608	Acceptable

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Product: PCB's in Water **WP Lot Number: 32705-32706** **APG+ Lot Number: 32756**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Aroclor 1016/1242 Sample 1	WP		<0.2					EPA 608	Acceptable
Aroclor 1016/1242 Sample 2	WP		<0.2					EPA 608	Acceptable
Aroclor 1221 Sample 1	WP	8885	<0.2					EPA 608	Acceptable
Aroclor 1232 Sample 1	WP	8890	<0.2					EPA 608	Acceptable
Aroclor 1232 Sample 2	WP	8890	<0.2					EPA 608	Acceptable
Aroclor 1248 Sample 1	WP	8900	<0.2					EPA 608	Acceptable
Aroclor 1248 Sample 2	WP	8900	<0.2					EPA 608	Acceptable
Aroclor 1254 Sample 1	WP	8905	<0.2					EPA 608	Acceptable
Aroclor 1254 Sample 2	WP	8905	3.93	5	2.37-6.48	0.714		EPA 608	Acceptable
Aroclor 1260 Sample 1	WP	8910	1.79	1.79	0.735-2.41	0.786		EPA 608	Acceptable
Aroclor 1260 Sample 2	WP	8910	<0.2					EPA 608	Acceptable

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Product: Pesticides (NELAC) **WP Lot Number: 32729**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
alpha-BHC	WP	7110	12.8	13.3	4.62-18.6	0.515		EPA 608	Acceptable
beta-BHC	WP	7115	8.31	8.16	3.01-11.8	0.623		EPA 608	Acceptable
delta-BHC	WP	7105	39.5	29.3	6.59-42.1	2.55		EPA 608	Check for Error
Endosulfan I	WP	7510	5.59	5.17	2.12-7.35	0.975		EPA 608	Acceptable
Endosulfan II	WP	7515	26.6	26.8	5.29-43	0.398		EPA 608	Acceptable
Endosulfan sulfate	WP	7520	17.3	17.7	3.08-29	0.301		EPA 608	Acceptable
Endrin	WP	7540	6.31	4.97	2.04-6.82	2.36		EPA 608	Check for Error
Endrin aldehyde	WP	7530	5.46	5.93	1.95-8.71	0.115		EPA 608	Acceptable
gamma-BHC	WP	7120	21.6	19.7	6.19-28.6	1.12		EPA 608	Acceptable
Methoxychlor	WP	7810	3.97	4.88	1.74-7.61	0.726		EPA 608	Acceptable

Product: Pesticides (WP) **WP Lot Number: 32728**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
4,4'-DDD	WP	7355	1.43	0.822	0.391-1.16	5.12		EPA 608	**Not Acceptable**
4,4'-DDE	WP	7360	2.80	2.62	1.24-3.58	1		EPA 608	Acceptable
4,4'-DDT	WP	7365	4.02	4.53	2.07-6.13	0.118		EPA 608	Acceptable
Aldrin	WP	7025	0.107	0.15	0.0346-0.206	0.456		EPA 608	Acceptable
Dieldrin	WP	7470	0.430	0.43	0.219-0.598	0.349		EPA 608	Acceptable
Heptachlor	WP	7685	1.23	1.32	0.352-1.84	0.567		EPA 608	Acceptable
Heptachlor epoxide (beta)	WP	7690	0.384	0.335	0.168-0.435	1.85		EPA 608	Acceptable

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Product: pH **WP Lot Number: 32698** **APG+ Lot Number: 32736**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
pH	WP	1900	5.75	5.83	5.7-5.96	1.82	10008205	EPA 150.1	Acceptable

Product: Polynuclear Aromatic Hydrocarbons-PAH **WP Lot Number: 32715** **APG+ Lot Number: 32751**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Acenaphthene	WP	5500	5.20	5.64	1.44-6.69	1.3		EPA 625 - SIM	Acceptable
Acenaphthylene	WP	5505	3.36	2.84	0.0064-4.23	1.76		EPA 625 - SIM	Acceptable
Anthracene	WP	5555	0.48	0.466	0.118-0.586	1.64		EPA 625 - SIM	Acceptable
Benzo(a)anthracene	WP	5575	<0.2					EPA 625 - SIM	Acceptable
Benzo(a)pyrene	WP	5580	<0.2					EPA 625 - SIM	Acceptable
Benzo(b)fluoranthene	WP	5585	<0.2					EPA 625 - SIM	Acceptable
Benzo(g,h,i)perylene	WP	5590	<0.2					EPA 625 - SIM	Acceptable
Benzo(k)fluoranthene	WP	5600	<0.2					EPA 625 - SIM	Acceptable
Chrysene	WP	5855	<0.2					EPA 625 - SIM	Acceptable
Dibenzo(a,h)anthracene	WP	5895	<0.2					EPA 625 - SIM	Acceptable
Fluoranthene	WP	6265	1.89	1.74	0.652-2.23	1.72		EPA 625 - SIM	Acceptable
Fluorene	WP	6270	<0.2					EPA 625 - SIM	Acceptable
Indeno(1,2,3-cd)pyrene	WP	6315	<0.2					EPA 625 - SIM	Acceptable
Naphthalene	WP	5005	16.0	22	6.54-23	0.438		EPA 625	Acceptable
Phenanthrene	WP	6615	<0.2					EPA 625 - SIM	Acceptable
Pyrene	WP	6665	0.81	0.735	0.215-1.14	0.864		EPA 625 - SIM	Acceptable

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Product: Solids **WP Lot Number: 77489**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Dissolved Solids	WP	1705	247	239	206-278	0.417	10009208	EPA 160.1	Acceptable
Total Solids	WP		324	319	284-358	0.244		EPA 160.3	Acceptable
Total Suspended Solids	WP		77	79.7	61.6-86	0.786		EPA 160.2	Acceptable

Product: Total Cyanide **WP Lot Number: 32702** **APG+ Lot Number: 32740**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Cyanide	WP	1645	0.513	0.507	0.349-0.654	0.217	10060205	EPA 335.2	Acceptable

Product: Total Phenolics **WP Lot Number: 32701** **APG+ Lot Number: 32739**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Phenolics	WP	1905	1.11	2.39	0.874-2.29	1.99		EPA 420.2	Acceptable

Product: Total Residual Chlorine **WP Lot Number: 32703** **APG+ Lot Number: 32741**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Residual Chlorine	WP	1940	1.3	1.25	0.966-1.49	0.796	10059004	EPA 330.4	Acceptable

Product: Toxaphene **WP Lot Number: 32719** **APG+ Lot Number: 32757**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Toxaphene	WP	8250	8.42	7.07	2.14-11.2	1.14		EPA 608	Acceptable

APG

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Product: TPH Diesel (DRO) **WP Lot Number: 32717** **APG+ Lot Number: 32754**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
TPH Diesel (DRO)	WP	9369	2.26	3.82	0-5.86	0.419		OA-2, 8015 mod.	Acceptable

Product: TPH Gasoline (GRO) **WP Lot Number: 32718** **APG+ Lot Number: 32753**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
TPH Gasoline (GRO)	WP	9408	1.83	2.08	1.02-3.04	0.595		OA1-8015mod.	Acceptable

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Product: Trace Metals **WP Lot Number: 32699-32700** **APG+ Lot Number: 32737-32738**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Aluminum	WP	1000	982	1000	852-1150	0.348	10014401	EPA 200.8	Acceptable
Antimony	WP	1005	223	231	154-282	0.236	10014401	EPA 200.8	Acceptable
Arsenic	WP	1010	298	303	252-356	0.345	10014401	EPA 200.8	Acceptable
Barium	WP	1015	1010	1050	896-1190	0.609	10014401	EPA 200.8	Acceptable
Beryllium	WP	1020	90.9	100	83.8-113	1.56	10014401	EPA 200.8	Acceptable
Boron	WP	1025		656	543-790				
Cadmium	WP	1030	173	178	151-203	0.463	10014401	EPA 200.8	Acceptable
Chromium	WP	1040	363	348	302-395	0.974	10014401	EPA 200.8	Acceptable
Cobalt	WP	1050	478	450	395-505	1.53	10014401	EPA 200.8	Acceptable
Copper	WP	1055	176	162	144-181	2.32	10014401	EPA 200.8	Check for Error
Iron	WP	1070	646	625	550-709	0.606	10013602	EPA 200.7	Acceptable
Lead	WP	1075	806	805	705-902	0.0912	10014401	EPA 200.8	Acceptable
Manganese	WP	1090	1675	1500	1350-1670	3.11	10014401	EPA 200.8	**Not Acceptable**
Mercury	WP	1095	7.56	8.16	6.06-10.2	0.848	10014401	EPA 200.8	Acceptable
Molybdenum	WP	1100	261	258	220-296	0.238	10014401	EPA 200.8	Acceptable
Nickel	WP	1105	2075	1890	1720-2110	2.54	10014401	EPA 200.8	Check for Error
Selenium	WP	1140	504	502	397-582	0.487	10014401	EPA 200.8	Acceptable
Silver	WP	1150	257	262	225-300	0.397	10014401	EPA 200.8	Acceptable
Strontium	WP		244	261	222-299	1.32		EPA 200.8	Acceptable
Thallium	WP	1165	237	241	192-282	0	10014401	EPA 200.8	Acceptable
Tin	WP	1175	2564	2700	2130-3290	0.756	10014401	EPA 200.8	Acceptable
Titanium	WP	1180	116	115	98-130	0.37		EPA 200.8	Acceptable
Vanadium	WP	1185	1052	1000	902-1100	1.58	10014401	EPA 200.8	Acceptable

APG Customer 3655
EPA Lab Code KS00902

Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

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Product: Trace Metals **WP Lot Number: 32699-32700** **APG+ Lot Number: 32737-32738**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Zinc	WP	1190	110	100	84.5-117	1.65	10014401	EPA 200.8	Acceptable

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Product: Volatiles **WP Lot Number: 32711** **APG+ Lot Number: 32747**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
1,1,1-Trichloroethane	WP	5160	48.8	47.3	30.6-62.4	0.434	10107207	EPA 624	Acceptable
1,1,2,2-Tetrachloroethane	WP	5110	146	128	75.4-179	1.1	10107207	EPA 624	Acceptable
1,1,2-Trichloroethane	WP	5165	116	138	93.2-180	1.39	10107207	EPA 624	Acceptable
1,1-Dichloroethane	WP	4630	<2.0				10107207	EPA 624	Acceptable
1,1-Dichloroethene	WP	4640	38.0	37.5	19.6-58.7	0.184	10107207	EPA 624	Acceptable
1,2-Dichloroethane	WP	4635	19.4	21.7	15.2-29.4	1.22	10107207	EPA 624	Acceptable
1,2-Dichloropropane	WP	4655	61.6	70.6	48-89.7	1.04	10107207	EPA 624	Acceptable
Bromodichloromethane	WP	4395	28.5	32.3	22.8-42.1	1.24	10107207	EPA 624	Acceptable
Bromoform	WP	4400	30.2	37.7	24.2-51.7	1.7	10107207	EPA 624	Acceptable
Bromomethane	WP	4950	35.5	44	11.6-77.1	0.807	10107207	EPA 624	Acceptable
Carbon tetrachloride	WP	4455	38.5	39	24.2-55	0.214	10107207	EPA 624	Acceptable
Chlorobenzene	WP	4475	19.6	21.2	15.4-26.8	0.789	10107207	EPA 624	Acceptable
Chloroethane	WP	4485	<2.0				10107207	EPA 624	Acceptable
Chloroform	WP	4505	44.2	45.9	31.9-58.9	0.267	10107207	EPA 624	Acceptable
Chloromethane	WP	4960	<2.0				10107207	EPA 624	Acceptable
Dibromochloromethane	WP	4575	9.84	12	7.85-15.8	1.47	10107207	EPA 624	Acceptable
Methylene chloride	WP	4975	45.6	46.6	29.4-64.3	0.224	10107207	EPA 624	Acceptable
Tetrachloroethene	WP	5115	23.2	25	16.5-32.3	0.455	10107207	EPA 624	Acceptable
trans-1,2-Dichloroethene	WP	4700	<2.0				10107207	EPA 624	Acceptable
trans-1,3-Dichloropropene	WP	4685	<2.0				10107207	EPA 624	Acceptable
Trichloroethene	WP	5170	45.6	50.7	32.9-65.5	0.662	10107207	EPA 624	Acceptable
Trichlorofluoromethane	WP	5175	<2.0				10107207	EPA 624	Acceptable
Vinyl Chloride	WP	5235	<2.0				10107207	EPA 624	Acceptable

Analytical Products Group, Inc.

PERFORMANCE REPORT

WP Performance Summary

October 2002

APG Customer Code: 3655

Analytical Management Lab
Suite B
15130 South Keeler
Olathe, KS 66062

Laboratory of
EXCELLENCE

NVLAP®

APG Customer 3655 Analytical Management Lab
EPA Lab Code KS00902 15130 South Keeler
Olathe, KS 66062

Performance Summary

WP October 2002
Study Closing Date 11/15/2002

Product: Acid Extractables **WP Lot Number: 34013** **APG+ Lot Number: 34065**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
2,4,5-Trichlorophenol	WP	6835	127	175	61.9-223	0.56	10107401	EPA 625	Acceptable
2,4,6-Trichlorophenol	WP	6840	91.4	123	40.2-163	0.517	10107401	EPA 625	Acceptable
2,4-Dichlorophenol	WP	6000	110	169	60.8-202	0.894	10107401	EPA 625	Acceptable
2,4-Dimethylphenol	WP	6130	79.9	135	29.6-177	0.943	10107401	EPA 625	Acceptable
2,4-Dinitrophenol	WP	6175	123	176	0-222	0.44	10107401	EPA 625	Acceptable
2-Chlorophenol	WP	5800	21.5	37.3	12.6-48	1.49	10107401	EPA 625	Acceptable
2-Methyl-4,6-Dinitrophenol	WP	6360	102	152	16.8-221	0.5	10107401	EPA 625	Acceptable
2-Nitrophenol	WP	6490	64.7	99.8	30-131	0.935	10107401	EPA 625	Acceptable
4-Chloro-3-methylphenol	WP	5700	124	179	70.8-230	0.981	10107401	EPA 625	Acceptable
4-Nitrophenol	WP	6500	11.8	17.9	0-33.1	0.0822	10107401	EPA 625	Acceptable
Pentachlorophenol	WP	6605	18.4	21.1	0.474-29.1	0.755	10107401	EPA 625	Acceptable
Phenol	WP	6625	29.6	83.1	0-113	0.783	10107401	EPA 625	Acceptable

Product: Aluminum High Level **WP Lot Number: 34025** **APG+ Lot Number: 34059**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Aluminum High Level	WP		0.686	0.7	0.628-0.774	0.612	10014003	EPA 200.7	Acceptable

APG Customer 3655
EPA Lab Code KS00902

Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Performance Summary

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Product: Aromatic Hydrocarbons **WP Lot Number: 34029** **APG+ Lot Number: 34078**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
1,2-Dichlorobenzene	WP	4610	13.0	13.4	9.13-17.5	0.214	10107207	EPA 624	Acceptable
1,3-Dichlorobenzene	WP	4615	30.0	31.3	22-39.2	0.209	10107207	EPA 624	Acceptable
1,4-Dichlorobenzene	WP	4620	32.6	33.2	22.8-43	0.089	10107207	EPA 624	Acceptable
Benzene	WP	4375	58.1	59.4	42.9-76.3	0.269	10107207	EPA 624	Acceptable
Ethylbenzene	WP	4765	30.5	30	20.2-39.1	0.285	10107207	EPA 624	Acceptable
m-Xylene / p-Xylene	WP	5240	116	124	85.5-162	0.625	10107207	EPA 624	Acceptable
o-Xylene	WP	5250	66.3	70.1	45.9-93.6	0.428	10107207	EPA 624	Acceptable
Toluene	WP	5140	57.6	58.9	42.4-73.4	0.0581	10107207	EPA 624	Acceptable
Total Xylenes	WP	5260	174	194	112-259	0.447	10107207	EPA 624	Acceptable

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Product: Base Neutral Extractables **WP Lot Number: 34984** **APG+ Lot Number: 34066**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
1,2,4-Trichlorobenzene	WP	5155	64.1	115	33.6-137	1.23	10107401	EPA 625	Acceptable
1,2-Dichlorobenzene	WP	4610	44.7	85.8	24.5-95.9	1.3	10107401	EPA 625	Acceptable
1,3-Dichlorobenzene	WP	4615	<10				10107401	EPA 625	Acceptable
1,4-Dichlorobenzene	WP	4620	<10				10107401	EPA 625	Acceptable
2,4-Dinitrotoluene (2,4-DNT)	WP	6185	115	154	65.7-205	0.901	10107401	EPA 625	Acceptable
2,6-Dinitrotoluene (2,6-DNT)	WP	6190	141	186	71.1-247	0.612	10107401	EPA 625	Acceptable
2-Chloronaphthalene	WP	5795	117	185	74.8-206	1.1	10107401	EPA 625	Acceptable
3,3'-Dichlorobenzidine	WP	5945	<10				10107401	EPA 625	Acceptable
4-Bromophenyl phenyl ether	WP	5660	<10				10107401	EPA 625	Acceptable
4-Chlorophenyl phenyl ether	WP	5825	<10				10107401	EPA 625	Acceptable
Acenaphthene	WP	5500	127	193	70.7-246	1.09	10107401	EPA 625	Acceptable
Acenaphthylene	WP	5505	97.5	119	47.5-144	0.106	10107401	EPA 625	Acceptable
Anthracene	WP	5555	29.7	40.7	18.5-52.7	1.04	10107401	EPA 625	Acceptable
Benzidine	WP	5595	<10				10107401	EPA 625	Acceptable
Benzo(a)anthracene	WP	5575	130	161	79.6-201	0.493	10107401	EPA 625	Acceptable
Benzo(a)pyrene	WP	5580	68.7	69.4	16.9-90.5	1.22	10107401	EPA 625	Acceptable
Benzo(b)fluoranthene	WP	5585	<10				10107401	EPA 625	Acceptable
Benzo(g,h,i)perylene	WP	5590	<10				10107401	EPA 625	Acceptable
Benzo(k)fluoranthene	WP	5600	<10				10107401	EPA 625	Acceptable
Bis(2-Chloroethoxy)methane	WP	5760	<10				10107401	EPA 625	Acceptable
Bis(2-Chloroethyl)ether	WP	5765	<10				10107401	EPA 625	Acceptable
Bis(2-Chloroisopropyl)ether	WP	5780	<10				10107401	EPA 625	Acceptable
Bis(2-Ethylhexyl)phthalate	WP	6255	51.1	61.6	17-89.4	0.174	10107401	EPA 625	Acceptable

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Product: Base Neutral Extractables **WP Lot Number: 34984** **APG+ Lot Number: 34066**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Butyl benzyl phthalate	WP	5670	125	172	58-224	0.58	10107401	EPA 625	Acceptable
Chrysene	WP	5855	58.6	72	28.4-94.4	0.255	10107401	EPA 625	Acceptable
Dibenzo(a,h)anthracene	WP	5895	<10				10107401	EPA 625	Acceptable
Diethyl phthalate	WP	6070	41.6	62.4	5.66-94.9	0.584	10107401	EPA 625	Acceptable
Dimethyl phthalate	WP	6135	92.3	178	0-273	0.325	10107401	EPA 625	Acceptable
Di-n-butyl phthalate	WP	5925	132	173	16.9-241	0.0802	10107401	EPA 625	Acceptable
Di-n-octyl phthalate	WP	6200	115	129	17.9-187	0.426	10107401	EPA 625	Acceptable
Fluoranthene	WP	6265	126	166	69.5-215	0.661	10107401	EPA 625	Acceptable
Fluorene	WP	6270	112	163	68.1-208	1.12	10107401	EPA 625	Acceptable
Hexachlorobenzene	WP	6275	137	188	82.4-242	0.94	10107401	EPA 625	Acceptable
Hexachlorobutadiene	WP	4835	41.6	73	11.6-88.6	0.664	10107401	EPA 625	Acceptable
Hexachlorocyclopentadiene	WP	6285	112	131	0-167	1.19	10107401	EPA 625	Acceptable
Hexachloroethane	WP	4840	87.6	171	13.1-218	0.83	10107401	EPA 625	Acceptable
Indeno(1,2,3-cd)pyrene	WP	6315	<10				10107401	EPA 625	Acceptable
Isophorone	WP	6320	58.5	68.5	25.1-90.4	0.0642	10107401	EPA 625	Acceptable
Naphthalene	WP	5005	60.8	105	26.5-137	1.14	10107401	EPA 625	Acceptable
Nitrobenzene (NB)	WP	5015	62.4	106	34.6-138	1.39	10107401	EPA 625	Acceptable
N-nitrosodimethylamine	WP	6530	<10				10107401	EPA 625	Acceptable
N-nitrosodi-n-propylamine	WP	6545	<10				10107401	EPA 625	Acceptable
N-nitrosodiphenylamine	WP	6535	<10				10107401	EPA 625	Acceptable
Phenanthrene	WP	6615	<10				10107401	EPA 625	Acceptable
Pyrene	WP	6665	137	182	60.8-243	0.493	10107401	EPA 625	Acceptable

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Product: BTEX **WP Lot Number: 34016** **APG+ Lot Number: 34068**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Benzene	WP	4375	20.2	20.8	14.7-27.1	0.338	10107207	EPA 624	Acceptable
Ethylbenzene	WP	4765	13.0	13.2	9.08-17	0	10107207	EPA 624	Acceptable
m-Xylene / p-Xylene	WP	5240	87.7	97.2	69-115	0.563	10107207	EPA 624	Acceptable
o-Xylene	WP	5250	121	131	94.6-161	0.636	10107207	EPA 624	Acceptable
Toluene	WP	5140	25.9	27	19.2-34	0.283	10107207	EPA 624	Acceptable
Total Xylenes	WP	5260	209	228	131-304	0.312	10107207	EPA 624	Acceptable

Product: Chlordane (Total) **WP Lot Number: 34010** **APG+ Lot Number: 34075**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Chlordane (Total)	WP	7250	10.2	11.5	5.02-16.4	0.265	10103603	EPA 608	Acceptable

Product: Demand **WP Lot Number: 33994** **APG+ Lot Number: 34047**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Biochemical Oxygen Demand	WP	1530	19.1	21.6	10.6-32.6	0.682	20027401	SM 18th ED 5210	Acceptable
Carbonaceous BOD	WP	1555	20.6	18.7	8.35-29	0.551	20027401	SM 18th ED 5210	Acceptable
Chemical Oxygen Demand	WP	1565	39.6	34.7	21.2-45.8	1.49	10076003	EPA 410.1	Acceptable
Total Organic Carbon	WP	2040		13.7	11.3-16.2				

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Product: Fluoride **WP Lot Number: 34026** **APG+ Lot Number: 34060**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Fluoride	WP	1730	2.24	2.25	1.96-2.52	0	10053006	EPA 300.0	Acceptable

Product: Herbicides **WP Lot Number: 34028** **APG+ Lot Number: 34076**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
2,4,5-T	WP	8655	0.713	2.64	0-4.17	1.92	10087204	EPA 515.1	Acceptable
2,4,5-TP (silvex)	WP	8650	2.53	6.67	0.343-8.97	1.48	10087204	EPA 515.1	Acceptable
2,4-D	WP	8545	1.40	5.4	0-8.51	1.82	10087204	EPA 515.1	Acceptable
Dicamba	WP	8595	1.70	7.33	0.63-10.6	2.36	10087204	EPA 515.1	Check for Error
Dinoseb	WP	8620	0.71	8.63	3.47-11.2	5.14	10087204	EPA 515.1	**Not Acceptable**

Product: Hexavalent Chromium **WP Lot Number: 34012** **APG+ Lot Number: 34062**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Chromium, Hexavalent	WP		342	334	272-384	0.749	10162206	EPA 7196	Acceptable

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Product: Minerals **WP Lot Number: 33997-33998** **APG+ Lot Number: 34051-34052**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Alkalinity as CaCO3	WP	1505	90.1	89	80.6-96.9	0.48	10054805	EPA 310.1	Acceptable
Calcium	WP	1035	26.7	26.4	23.4-30.1	0.0885	10014003	EPA 200.7	Acceptable
Chloride	WP	1575	173	174	159-189	0.2	10053006	EPA 300.0	Acceptable
Conductivity	WP	1610	778	784	718-848	0.233	10006209	EPA 120.1	Acceptable
Magnesium	WP	1085	11.1	10.8	9.39-12.2	0.651	10014003	EPA 200.7	Acceptable
Potassium	WP	1125	30.0	31.5	27.2-35.9	1.1	10014003	EPA 200.7	Acceptable
Sodium	WP	1155	91.0	90.8	82.1-99.3	0.105	10014003	EPA 200.7	Acceptable
Sulfate	WP	2000	12.3	13.4	9.97-16.5	0.917	10053006	EPA 300.0	Acceptable
Total Dissolved Solids	WP	1705	404	413	307-499	0.0312	10009208	EPA 160.1	Acceptable
Total Hardness as CaCO3	WP	1755	112	111	100-123	0.265	20003401	SM 18/19thED 23	Acceptable

Product: Nutrient **WP Lot Number: 33995-33996** **APG+ Lot Number: 34048-34049**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Ammonia Nitrogen as N	WP	1515	11.4	9.89	7.66-12	2.14	10064401	EPA 350.3	Check for Error
Nitrate Nitrogen as N	WP	1810	5.06	5.36	4.22-6.4	0.689	10053006	EPA 300.0	Acceptable
Orthophosphate as P	WP	1870	2.65	2.64	2.25-3.06	0	10053006	EPA 300.0	Acceptable
Total Kjeldahl Nitrogen	WP	1795		34.2	25.2-41.9				
Total Phosphorus as P	WP	1910	1.01	5.32	4.04-6.23	11.3	10070801	EPA 365.3	**Not Acceptable**

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Product: Oil & Grease **WP Lot Number: 34005** **APG+ Lot Number: 34050**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Oil & Grease	WP	1860	27.2	23	13.8-27.9	2.67	10127409	EPA 1664	Check for Error

Product: PCB's in Oil **WP Lot Number: 34008-34009**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Aroclor 1016/1242 in Oil Sample 1	WP		<10				10103603	EPA 608	Acceptable
Aroclor 1016/1242 in Oil Sample 2	WP		29.5	42.2	7.85-56.4	0.321	10103603	EPA 608	Acceptable
Aroclor 1254 in Oil Sample 1	WP	8905	13.6	16.3	0.0513-26.9	0.0223	10103603	EPA 608	Acceptable
Aroclor 1254 in Oil Sample 2	WP	8905	<10				10103603	EPA 608	Acceptable
Aroclor 1260 in Oil Sample 1	WP	8910	<10				10103603	EPA 608	Acceptable
Aroclor 1260 in Oil Sample 2	WP	8910	<10				10103603	EPA 608	Acceptable

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Product: PCB's in Water **WP Lot Number: 34006-34007** **APG+ Lot Number: 34072**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Aroclor 1016/1242 Sample 1	WP		7.54	8.5	2.39-12	0.233	10103603	EPA 608	Acceptable
Aroclor 1016/1242 Sample 2	WP		<1				10103603	EPA 608	Acceptable
Aroclor 1232 Sample 1	WP	8890	<1				10103603	EPA 608	Acceptable
Aroclor 1232 Sample 2	WP	8890	<1				10103603	EPA 608	Acceptable
Aroclor 1248 Sample 1	WP	8900	<1				10103603	EPA 608	Acceptable
Aroclor 1248 Sample 2	WP	8900	<1				10103603	EPA 608	Acceptable
Aroclor 1254 Sample 1	WP	8905	<1				10103603	EPA 608	Acceptable
Aroclor 1254 Sample 2	WP	8905	1.89	2.06	0.828-2.95	0	10103603	EPA 608	Acceptable
Aroclor 1260 Sample 1	WP	8910	<1				10103603	EPA 608	Acceptable
Aroclor 1260 Sample 2	WP	8910	<1				10103603	EPA 608	Acceptable

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Product: Pesticides (NELAC) **WP Lot Number: 34031**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
alpha-BHC	WP	7110	11.7	13.3	4.62-18.6	0.0429	10103603	EPA 608	Acceptable
beta-BHC	WP	7115	7.78	8.51	3.14-12.3	0.0461	10103603	EPA 608	Acceptable
delta-BHC	WP	7105	13.7	14.6	3.83-19.9	0.672	10103603	EPA 608	Acceptable
Endosulfan I	WP	7510	6.87	9.57	3.95-13.9	1.23	10103603	EPA 608	Acceptable
Endosulfan II	WP	7515	37.0	37.4	6.86-60.5	0.369	10103603	EPA 608	Acceptable
Endosulfan sulfate	WP	7520	17.5	20.6	3.39-33.9	0.217	10103603	EPA 608	Acceptable
Endrin	WP	7540	14.2	15.2	5.39-22.7	0.0694	10103603	EPA 608	Acceptable
Endrin aldehyde	WP	7530	11.8	14.2	4.64-20.8	0.335	10103603	EPA 608	Acceptable
gamma-BHC	WP	7120	3.46	3.71	1.36-5.29	0.214	10103603	EPA 608	Acceptable
Methoxychlor	WP	7810	17.6	18	7.59-25.3	0.407	10103603	EPA 608	Acceptable

Product: Pesticides (WP) **WP Lot Number: 34030**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
4,4'-DDD	WP	7355	1.01	0.841	0.4-1.19	1.66	10103603	EPA 608	Acceptable
4,4'-DDE	WP	7360	0.562	0.431	0.194-0.586	2.63	10103603	EPA 608	Check for Error
4,4'-DDT	WP	7365	0.487	0.438	0.182-0.622	1.16	10103603	EPA 608	Acceptable
Aldrin	WP	7025	0.818	1.1	0.279-1.49	0.327	10103603	EPA 608	Acceptable
Dieldrin	WP	7470	0.509	0.423	0.215-0.588	1.72	10103603	EPA 608	Acceptable
Heptachlor	WP	7685	1.90	2.19	0.579-3.05	0.218	10103603	EPA 608	Acceptable
Heptachlor epoxide (beta)	WP	7690	1.68	1.73	0.879-2.23	0.58	10103603	EPA 608	Acceptable

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Product: pH **WP Lot Number: 33999** **APG+ Lot Number: 34053**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
pH	WP	1900	7.45	7.44	7.24-7.64	0.149	10008409	EPA 150.1	Acceptable

Product: Polynuclear Aromatic Hydrocarbons-PAH **WP Lot Number: 34015** **APG+ Lot Number: 34067**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Acenaphthene	WP	5500	<0.2				10107401	EPA 625 SIM	Acceptable
Acenaphthylene	WP	5505	<0.2				10107401	EPA 625 SIM	Acceptable
Anthracene	WP	5555	<0.2				10107401	EPA 625 SIM	Acceptable
Benzo(a)anthracene	WP	5575	2.00	1.85	1.06-2.34	1.4	10107401	EPA 625 SIM	Acceptable
Benzo(a)pyrene	WP	5580	1.80	1.24	0-2.49	1.39	10107401	EPA 625 SIM	Acceptable
Benzo(b)fluoranthene	WP	5585	<0.2				10107401	EPA 625 SIM	Acceptable
Benzo(g,h,i)perylene	WP	5590	<0.2				10107401	EPA 625 SIM	Acceptable
Benzo(k)fluoranthene	WP	5600	<0.2				10107401	EPA 625 SIM	Acceptable
Chrysene	WP	5855	2.30	1.89	1.03-2.49	2.21	10107401	EPA 625 SIM	Check for Error
Dibenzo(a,h)anthracene	WP	5895	<0.2				10107401	EPA 625 SIM	Acceptable
Fluoranthene	WP	6265	1.02	0.824	0.263-1.26	1.54	10107401	EPA 625 SIM	Acceptable
Fluorene	WP	6270	9.41	8.78	4.88-8.73	4.07	10107401	EPA 625 SIM	**Not Acceptable**
Indeno(1,2,3-cd)pyrene	WP	6315	<0.2				10107401	EPA 625 SIM	Acceptable
Naphthalene	WP	5005	6.22	7.36	3.46-7.66	0.943	10107401	EPA 625 SIM	Acceptable
Phenanthrene	WP	6615	<0.2				10107401	EPA 625 SIM	Acceptable
Pyrene	WP	6665	1.71	1.53	0.744-2.14	1.16	10107401	EPA 625 SIM	Acceptable

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Product: Solids **WP Lot Number: 80999**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Dissolved Solids	WP		123	229	163-309	4.67	10009208	EPA 160.1	Not Acceptable
Total Solids	WP		188	302	249-355	6.4		EPA 160.3	Not Acceptable
Total Suspended Solids	WP		68	73.4	56.5-79.1	0.0531	10009606	EPA 160.2	Acceptable

Product: Total Cyanide **WP Lot Number: 34003** **APG+ Lot Number: 34057**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Cyanide	WP	1645	0.61	0.897	0.624-1.15	3.17	10060409	EPA 335.2	Not Acceptable

Product: Total Phenolics **WP Lot Number: 34002** **APG+ Lot Number: 34056**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Phenolics	WP	1905	1.74	1.78	0.648-1.71	3.16	10079808	EPA 420.2	Not Acceptable

Product: Total Residual Chlorine **WP Lot Number: 34004** **APG+ Lot Number: 34882**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Residual Chlorine	WP	1940	3.1	3.43	2.62-3.76	0.474	10059208	EPA 330.4	Acceptable

Product: Toxaphene **WP Lot Number: 34019** **APG+ Lot Number: 34073**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Toxaphene	WP	8250	6.99	8.87	2.31-15.1	0.808	10103603	EPA 608	Acceptable

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Product: TPH Diesel (DRO) **WP Lot Number: 34017** **APG+ Lot Number: 34070**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
TPH Diesel (DRO)	WP	9369	0.715	1.03	0-264	0.243	10173203	EPA 8015	Acceptable

Product: TPH Gasoline (GRO) **WP Lot Number: 34018** **APG+ Lot Number: 34069**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
TPH Gasoline (GRO)	WP	9408	3.73	4.4	1.28-5.75	0.282	10173203	EPA 8015	Acceptable

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Product: Trace Metals **WP Lot Number: 34000-34001** **APG+ Lot Number: 34054-34055**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Aluminum	WP	1000	1619	1660	1420-1890	0.403	10014003	EPA 200.7	Acceptable
Antimony	WP	1005	192	203	134-248	0.0526	10014809	EPA 200.8	Acceptable
Arsenic	WP	1010	634	666	559-779	0.956	10014809	EPA 200.8	Acceptable
Barium	WP	1015	1751	1770	1500-2000	0.012	10014003	EPA 200.7	Acceptable
Beryllium	WP	1020	194	205	173-232	0.927	10014003	EPA 200.7	Acceptable
Boron	WP	1025	752	816	687-930	1.41	10014809	EPA 200.8	Acceptable
Cadmium	WP	1030	253	261	222-297	0.56	10014809	EPA 200.8	Acceptable
Chromium	WP	1040	248	245	212-278	0.27	10014809	EPA 200.8	Acceptable
Cobalt	WP	1050	745	708	622-793	1.3	10014003	EPA 200.7	Acceptable
Copper	WP	1055	167	169	151-188	0.321	10014003	EPA 200.7	Acceptable
Iron	WP	1070	506	500	439-569	0.093	10014003	EPA 200.7	Acceptable
Lead	WP	1075	1801	1760	1550-1970	0.594	10014003	EPA 200.7	Acceptable
Manganese	WP	1090	2780	2700	2430-3000	0.63	10014003	EPA 200.7	Acceptable
Mercury	WP	1095	7.56	8.13	6.03-10.2	0.807	10014809	EPA 200.8	Acceptable
Molybdenum	WP	1100	331	317	271-363	0.915	10014003	EPA 200.7	Acceptable
Nickel	WP	1105	307	283	250-319	1.91	10014809	EPA 200.8	Acceptable
Selenium	WP	1140	684	682	541-790	0.458	10014809	EPA 200.8	Acceptable
Silver	WP	1150	445	562	483-644	4.39	10014809	EPA 200.8	**Not Acceptable**
Strontium	WP		87.9	89.4	75.7-103	0.31	10014809	EPA 200.8	Acceptable
Thallium	WP	1165	482	482	388-560	0.279	10014809	EPA 200.8	Acceptable
Tin	WP	1175	1586	1530	1210-1860	0.519	10014809	EPA 200.8	Acceptable
Titanium	WP	1180	210	222	191-250	1.01	10014809	EPA 200.8	Acceptable
Vanadium	WP	1185	1314	1280	1160-1410	0.81	10014003	EPA 200.7	Acceptable

Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Performance Summary

WP October 2002
Study Closing Date 11/15/2002

Product: Trace Metals **WP Lot Number: 34000-34001** **APG+ Lot Number: 34054-34055**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Zinc	WP	1190	1609	1540	1370-1730	0.985	10014809	EPA 200.8	Acceptable

APG Customer 3655 Analytical Management Lab
EPA Lab Code KS00902 15130 South Keeler
Olathe, KS 66062

Performance Summary

WP October 2002
Study Closing Date 11/15/2002

Product: Volatiles **WP Lot Number: 35021** **APG+ Lot Number: 35022**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
1,1,1-Trichloroethane	WP	5160	54.2	59.2	38.2-78.2	0.601	10107207	EPA 624	Acceptable
1,1,2,2-Tetrachloroethane	WP	5110	147	140	82.5-195	0.426	10107207	EPA 624	Acceptable
1,1,2-Trichloroethane	WP	5165	107	121	81.9-157	1.03	10107207	EPA 624	Acceptable
1,1-Dichloroethane	WP	4630	<2				10107207	EPA 624	Acceptable
1,1-Dichloroethene	WP	4640	26.0	26	13-41.7	0.272	10107207	EPA 624	Acceptable
1,2-Dichloroethane	WP	4635	51.6	56.6	39.1-75.2	0.929	10107207	EPA 624	Acceptable
1,2-Dichloropropane	WP	4655	80.1	90.7	61.2-115	0.909	10107207	EPA 624	Acceptable
Bromodichloromethane	WP	4395	14.6	15.4	10.7-20.2	0.566	10107207	EPA 624	Acceptable
Bromoform	WP	4400	39.0	42.8	27.7-58.6	0.795	10107207	EPA 624	Acceptable
Bromomethane	WP	4950	<2				10107207	EPA 624	Acceptable
Carbon tetrachloride	WP	4455	24.4	24.1	15.1-34.1	0.0633	10107207	EPA 624	Acceptable
Chlorobenzene	WP	4475	13.2	13.4	10-16.9	0.175	10107207	EPA 624	Acceptable
Chloroethane	WP	4485	<2				10107207	EPA 624	Acceptable
Chloroform	WP	4505	48.3	50.1	34.8-64.3	0.244	10107207	EPA 624	Acceptable
Chloromethane	WP	4960	<2				10107207	EPA 624	Acceptable
Dibromochloromethane	WP	4575	20.0	21.4	14.1-28.4	0.504	10107207	EPA 624	Acceptable
Methylene chloride	WP	4975	18.4	19.1	12.1-26.9	0.445	10107207	EPA 624	Acceptable
Tetrachloroethene	WP	5115	54.0	55.4	36.7-70.9	0.035	10107207	EPA 624	Acceptable
trans-1,2-Dichloroethene	WP	4700	17.2	18.3	9.73-26.7	0.353	10107207	EPA 624	Acceptable
trans-1,3-Dichloropropene	WP	4685	<2				10107207	EPA 624	Acceptable
Trichloroethene	WP	5170	33.0	33.9	22.1-43.9	0	10107207	EPA 624	Acceptable
Trichlorofluoromethane	WP	5175	<2				10107207	EPA 624	Acceptable
Vinyl Chloride	WP	5235	<2				10107207	EPA 624	Acceptable

Analytical Products Group, Inc.

PERFORMANCE REPORT

WP Performance Summary

January 2003

APG Customer Code: 3655

Analytical Management Lab
Suite B
15130 South Keeler
Olathe, KS 66062

Laboratory of
EXCELLENCE

NVLAP®

APG Customer 3655 Analytical Management Lab
EPA Lab Code KS00902 15130 South Keeler
Olathe, KS 66062

Performance Summary

WP January 2003
Study Closing Date 02/19/2003

Product: Demand **WP Lot Number: 35239** **APG+ Lot Number: 35292**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Biochemical Oxygen Demand	WP	1530		115	58.2-172				
Carbonaceous BOD	WP	1555		98.6	44.2-153				
Chemical Oxygen Demand	WP	1565		185	143-210				
Total Organic Carbon	WP	2040	80.0	73.2	61.2-84.2	1.91	10078407	EPA415.1	Acceptable

Product: Solids **WP Lot Number: 82902**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Dissolved Solids	WP		75	85	58.6-112	1.16	10009208	EPA160.1	Acceptable
Total Solids	WP		132	164	127-193	2.52	10009800	EPA160.3	Check for Error
Total Suspended Solids	WP		63	79.5	61.4-85.8	2.61	10009606	EPA160.2	Check for Error

Product: Total Cyanide **WP Lot Number: 35248** **APG+ Lot Number: 35302**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Cyanide	WP	1645	0.485	0.466	0.32-0.602	0.512	10060409	EPA335.2	Acceptable

Product: Total Phenolics **WP Lot Number: 35247** **APG+ Lot Number: 35301**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Phenolics	WP	1905	0.531	0.911	0.328-0.878	0.784	10079808	EPA420.2	Acceptable

Analytical Products Group, Inc.

PERFORMANCE REPORT

WP Performance Summary

February 2003

APG Customer Code: 3655

Analytical Management Lab
Suite B
15130 South Keeler
Olathe, KS 66062

Laboratory of
EXCELLENCE

NVLAP®
LAB CODE 200384-0

Performance Summary

WP February 2003
Study Closing Date 03/20/2003

Product: Demand **WP Lot Number: 35618**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Biochemical Oxygen Demand	WP	1530		100	50.5-149				
Carbonaceous BOD	WP	1555		86.4	38.7-134				
Chemical Oxygen Demand	WP	1565		162	124-185				
Total Organic Carbon	WP	2040	71.7	64.2	53.6-73.9	2.37	10078407	EPA415.1	Check for Error

Product: Solids **WP Lot Number: 83880**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Dissolved Solids	WP		99	156	117-191	4.44	10009208	EPA160.1	**Not Acceptable**
Total Solids	WP		153	187	147-217	2.46	10009800	EPA160.3	Check for Error
Total Suspended Solids	WP		28	31.1	22.6-33	0.114	10009606	EPA160.2	Acceptable

Product: Total Cyanide **WP Lot Number: 35627**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Cyanide	WP	1645	0.568	0.549	0.379-0.708	0.456	10060409	EPA 335.2	Acceptable

Product: Total Phenolics **WP Lot Number: 35626**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Phenolics	WP	1905	0.076	0.142	0.0435-0.145	1.08	10079808	EPA420.2	Acceptable

Analytical Products Group, Inc.

PERFORMANCE REPORT

WP Performance Summary

April 2003

APG Customer Code: 3655

Analytical Management Lab
Suite B
15130 South Keeler
Olathe, KS 66062

Laboratory of
EXCELLENCE

NVLAP®
LAB CODE 200384-0

Water Pollution (WP) Proficiency Testing Program Report

Introduction:

The Analytical Products Group, Inc. Proficiency Testing Program has been designed to evaluate your laboratory's performance against other participating laboratories on the same set of standards. This report has been specifically prepared for your organization to assist you in ensuring that your procedures are in-control. Therefore, in most cases sample levels are not designed to challenge the detection limits of the method.

The Analytical Products Group Proficiency Testing Program is certified to meet all of the following requirements:

- Accredited by NIST according to the National Voluntary Laboratory Accreditation Program (Handbook 150-19) for Chemical Calibrations: Providers of Proficiency Testing (NVLAB Code 200384-0)
- Analytical Products Group, Inc. Quality System is registered with DNVCertification to meet the ISO 9001 standard to design, manufacture, and distribute chemical reference materials and operate Proficiency Testing Programs (Certificate # 98-HOU-AQ-9513)
- Proficiency Testing Program is certified to meet all requirements as specified in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).
- Certified to meet ISO/REMCO Harmonized Protocol for Proficiency Testing of Analytical Laboratories
- Certified to meet ASTM E1301

Report Layout

This report contains the following sub-reports:

Introduction -This section is the general introduction to the Analytical Products Group, Inc. Proficiency Testing Program as well as all of the certifications that the organization maintains.

Terms & Definitions - Defines all of the terms used throughout the report. This is an excellent section to refer to first if you have any questions.

Laboratory Benchmark Report - This report presents an overall summary of the study as well as your performance relative to all other study participants.

Comment Report - Contains comments submitted to Analytical Products Group by the program participants or comments by Analytical Products Group to program participants concerning unusual characteristics of the data set or problems with standards which could influence the interpretation of the data. Please review this section thoroughly before examining your data.

Exception Report - This report is a summary of all of the analytes that resulted in an Evaluation of "Not Acceptable." The Exception Report is designed to very quickly identify for your laboratory the areas that may require your immediate attention.

Performance Evaluation Summary Report - Provides a summary of all of your data for each analyte. This summary is formatted similar to the US EPA DMR-QA Reports.

Detail Report- This report lists all the details of your data. Also included in this section are useful graphs indicating the APG Assigned Value, the EPA Mean, the EPA Acceptance Limits and your Reported Value. Another graph included in this section shows your Z score for the analyte of interest.

The goal of this report is to provide your laboratory with the information you need to monitor and improve your quality. If you have any questions or feedback, please give us a call (800-272-4442). Thank you for choosing Analytical Products Group as your proficiency testing provider.

Sincerely,

Thomas J. Coyner
Proficiency Program Manager

Terms & Definitions

Average Acceptable Data - This value is the average percent of data that is within the EPA Acceptance Limits for this study. The Average Acceptable Data is reported for all laboratories as a whole as well as your laboratory as an individual. These values can be found in the Laboratory Benchmark Report.

Average Percentile for All Parameters - This is your laboratory's rank on each parameter and the related rank for that parameter. The average percentile is the average of this data across all parameters reported. It is a different measure of performance than percent acceptable data since it is affected by your relative performance on each parameter. The report provides an average for this measure for all laboratories reporting as well as your laboratory's performance.

Analyte - Name of the analyte of interest as found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) or NELAC Field of Testing List.

Analyte Code - This value is the code given to the analyte by the US EPA. These codes can be found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045). If no code is listed, then this analyte is not part of the US EPA program.

APG Assigned Value - This value is the calculated True Value of the standard based upon the actual composition of the standard.

APG Certified Value - This value is the result of the quality control testing performed on the sample by Analytical Products Group before the sample is shipped to the customer. Through this testing, the samples are traced against a NIST Standard Reference Material where available or an external standard if a NIST Standard Reference Material is not available. The reported uncertainty of this value is expressed as two times the standard deviation (s) divided by the square root of the number of samples (n) tested (i.e. 2s/vn). This quality control testing meets all of the requirements as specified by the NIST National Voluntary Laboratory Accreditation Program (NVLAP) Handbook 150-19 (Chemical Calibration: Providers of Proficiency Testing).

EPA Acceptance Range - This range is calculated based upon the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045). For the Water Pollution Program (WP), EPA Acceptance Limits are defined as ± three (3) EPA Standard Deviations from the EPA Mean.

EPA Mean - If the analyte is included in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then this value is the Mean Value calculated per the Criteria Document. If the analyte is not included in the Criteria Document, then this value is the Study Mean.

Terms & Definitions (continued)

Evaluation - This value is determined by the EPA Acceptance Limits and the Reported Value. Evaluation may have one of following values:

Value	Description
Acceptable	The Reported Value was within the EPA Acceptable Limits or a less than (<) value was reported AND zero (0) was included in the EPA Acceptance Limit Range AND the numeric part of the Reported Value was less than the upper limit of the EPA Acceptance Limit.
Not Acceptable	The Reported Value is outside of the EPA Acceptance Limits or a less than (<) value was reported AND zero (0) is not included in the EPA Acceptance Limits range or the numeric part of the Reported Value was greater than the upper limit of the EPA Acceptance Limit or a greater than value (>) was reported.
Check for Error	The Reported Value is between two (2) and three (3) EPA Standard Deviations from the EPA Mean.
Unusable	The Reported Value was a text value which could not be evaluated.
Blank	No data was reported for this analyte or the analyte was not spiked.

Inter-Laboratory Statistics - This data is a summary of all of the data from all study participants for the analyte of interest. Inter-Laboratory Statistics are only reported if a minimum number of laboratories analyzed and reported data for the analyte of interest.

Laboratory Pass Percentage - This is the percentage of study participants that reported values within the EPA Acceptance Limits for the analyte of interest.

Number of Acceptable Data Points - This value is the number of data points reported by your laboratory that were within the EPA Acceptance Limits.

Program - This value indicates which program the analyte of interest is a part. For Water Pollution Studies (WP), the only values that Program will have are 'WP' or 'APG+'. If the analyte and range are consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then the Program value will have a value of 'WP' and if this is not the case then the Program value will be 'APG+', indicating that the analyte and/or range was specified by Analytical Products Group.

Rank on Percent Acceptable Data - This is your laboratories rank out of all possible ranks in the current study based on percent acceptable data. It is important to note that several laboratories can achieve the same rank.

Rank on Average Percentile Data - This value is your laboratories rank out of all possible ranks based on Your Ranking converted to a percentile. It is important to note that several laboratories can achieve the same rank.

Reported Value - This value is the result that was reported by your organization for the particular analyte. Every effort has been made to ensure correct data entry. Should you find a data entry error, please notify Analytical Products Group, Inc. and we will correct the error and re-issue the report. If the Reported Value is reported as a less than (<) than figure, then the only way that the Evaluation will result in an "Acceptable" value is if the numeric part of the Reported Value (i.e. 8 if the Report Value were <8) is less then the upper limit of the EPA Acceptance Limit AND zero (0) is included in the EPA Acceptance Limit. Any other non-numeric value (i.e. BDL, ND, >10, etc.) cannot be evaluated and will return an Evaluation result of "Unusable." This method of evaluation is consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).

Solids Standards - Solids standards are individually prepared, therefore, each standard has a unique APG Assigned Value.

Study Mean - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Mean is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Terms & Definitions (continued)

Study Standard Deviation - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Standard Deviation is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Weighted Rank on Percent Acceptable Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percent Acceptable Data is the weighted rank of your laboratory based on Percent Acceptable Data and the number of analytes reported.

Weight Rank on Percentile Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percentile Data is the weighted rank of your laboratory based on Rank on Average Percentile Data and the number of analytes reported.

Your Ranking - This is your ranking based on your Z Score for the analyte of interest among all possible rankings. It is important to note that several laboratories can achieve the same rank.

Z-Score - This figure corresponds to how far your Reported Value was from the EPA Mean, reported in terms of EPA Standard Deviation units. As a point of reference, the US EPA Acceptance Limits are plus or minus three (3) EPA Standard Deviations from the EPA Mean.

Laboratory Benchmark Report

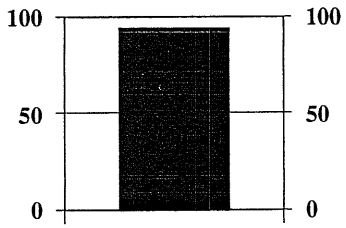
Customer Code: 3655 **WP April 2003**

Summary of Ranking Data

Summary of National Ranking Data

Total Number of Laboratories Reporting:	295
Average Percent Acceptable Data:	94.73%
Average Percentile for all Parameters:	53.69%

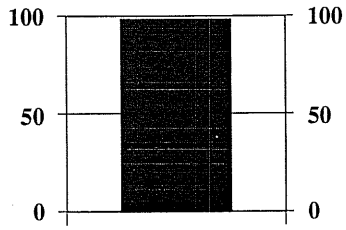
National Avg % Acceptable
94.7 %



Summary of Laboratory Ranking:

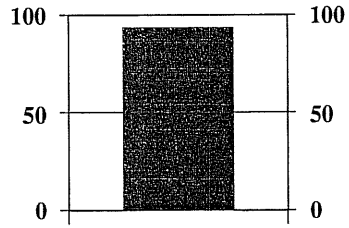
Total Data Points Reported:	197
Number of Acceptable Data Points:	194
Percent Acceptable Data:	98.48%
Average Percentile for all Parameters:	58.56%

Laboratory Avg % Acceptable
98.48%



Rank on Percent Acceptable Data:	13 of 78
Rank on Average Percentile Data:	109 of 254
Weighted Rank on Percent Acceptable Data:	19 of 122
Weighted Rank on Average Percentile Data:	17 of 265

Weighted Rank Avg Percentile
93.6 %



Comment Report

There are no comments to report.

APG Customer 3655 Analytical Management Lab
EPA Lab Code KS00902 15130 South Keeler
Olathe, KS 66062

Exception Report

WP April 2003
Study Closing Date 05/16/2003

Product: Solids **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Dissolved Solids	WP	1705	54.0	46.9	42.1-52.1	4.16	10009004	EPA160.1	Not Acceptable

Product: Trace Metals **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Nickel	WP	1105	1780	1590	1440-1770	3.09	10014003	EPA200.7	Not Acceptable

Product: Volatiles **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Trichloroethene	WP	5170	46.7	30.6	19.9-39.7	5.14	10107207	EPA624	Not Acceptable

Performance Summary

WP April 2003
Study Closing Date 05/16/2003

Product: Acid Extractables **WP Lot Number: 35911** **APG+ Lot Number: 35964**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
2,4,5-Trichlorophenol	WP	6835	138	171	60.6-218	0.0382	10107401	EPA625	Acceptable
2,4,6-Trichlorophenol	WP	6840	40.3	54.6	19-71.5	0.571	10107401	EPA625	Acceptable
2,4-Dichlorophenol	WP	6000	<5				10107401	EPA625	Acceptable
2,4-Dimethylphenol	WP	6130	5.2	31.2	3.83-42.6	2.78	10107401	EPA625	Check for Error
2,4-Dinitrophenol	WP	6175	79.9	151	0-194	0.39	10107401	EPA625	Acceptable
2-Chlorophenol	WP	5800	12.1	16.4	7.11-22	1	10107401	EPA625	Acceptable
2-Methyl-4,6-Dinitrophenol	WP	6360	45.7	81.3	16.5-109	1.11	10107401	EPA625	Acceptable
2-Nitrophenol	WP	6490	<5				10107401	EPA625	Acceptable
4-Chloro-3-methylphenol	WP	5700	134	182	72-234	0.704	10107401	EPA625	Acceptable
4-Nitrophenol	WP	6500	81.3	133	0-180	0.223	10107401	EPA625	Acceptable
Pentachlorophenol	WP	6605	18.1	18.5	0-25.5	1.28	10107401	EPA625	Acceptable
Phenol	WP	6625	18.3	27.8	0-40.3	0.273	10107401	EPA625	Acceptable

Product: Aluminum High Level **WP Lot Number: 35923** **APG+ Lot Number: 35958**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Aluminum High Level	WP	1000	0.856	0.8	0.69-0.885	2.13	10014003	EPA200.7	Check for Error

Performance Summary

Study Closing Date 05/16/2003

Product: Base Neutral Extractables **WP Lot Number: 35912** **APG+ Lot Number: 35965**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
1,2,4-Trichlorobenzene	WP	5155	41.3	55.6	16-68.3	0.0918	10107401	EPA625	Acceptable
1,2-Dichlorobenzene	WP	4610	30.1	42.3	12.3-47.5	0.0341	10107401	EPA625	Acceptable
1,3-Dichlorobenzene	WP	4615	60.9	84.8	14.4-101	0.229	10107401	EPA625	Acceptable
1,4-Dichlorobenzene	WP	4620	43.1	61.1	11.9-72.7	0.0792	10107401	EPA625	Acceptable
2,4-Dinitrotoluene (2,4-DNT)	WP	6185	52.4	71.8	27.7-95.1	0.804	10107401	EPA625	Acceptable
2,6-Dinitrotoluene (2,6-DNT)	WP	6190	<5				10107401	EPA625	Acceptable
2-Chloronaphthalene	WP	5795	130	165	66.6-185	0.203	10107401	EPA625	Acceptable
3,3'-Dichlorobenzidine	WP	5945	<5				10107401	EPA625	Acceptable
4-Bromophenyl phenyl ether	WP	5660	<5				10107401	EPA625	Acceptable
4-Chlorophenyl phenyl ether	WP	5825	<5				10107401	EPA625	Acceptable
Acenaphthene	WP	5500	125	157	57.8-200	0.168	10107401	EPA625	Acceptable
Acenaphthylene	WP	5505	78.0	99.7	39.6-121	0.169	10107401	EPA625	Acceptable
Anthracene	WP	5555	35.8	43.3	19.3-56.1	0.309	10107401	EPA625	Acceptable
Benzidine	WP	5595	<5				10107401	EPA625	Acceptable
Benzo(a)anthracene	WP	5575	65.5	80.3	41.6-98.4	0.476	10107401	EPA625	Acceptable
Benzo(a)pyrene	WP	5580	66.0	85.7	22.3-111	0.0541	10107401	EPA625	Acceptable
Benzo(b)fluoranthene	WP	5585	<5				10107401	EPA625	Acceptable
Benzo(g,h,i)perylene	WP	5590	<5				10107401	EPA625	Acceptable
Benzo(k)fluoranthene	WP	5600	<5				10107401	EPA625	Acceptable
Bis(2-Chloroethoxy)methane	WP	5760	<5				10107401	EPA625	Acceptable
Bis(2-Chloroethyl)ether	WP	5765	<5				10107401	EPA625	Acceptable
Bis(2-Chloroisopropyl)ether	WP	5780	<5				10107401	EPA625	Acceptable
Bis(2-Ethylhexyl)phthalate	WP	6255	38.4	56.4	16.8-81.7	1	10107401	EPA625	Acceptable
Butyl benzyl phthalate	WP	5670	134	161	0-251	0.545	10107401	EPA625	Acceptable
Chrysene	WP	5855	82.7	96.5	36.5-127	0.0596	10107401	EPA625	Acceptable
Dibenzo(a,h)anthracene	WP	5895	<5				10107401	EPA625	Acceptable
Diethyl phthalate	WP	6070	131	163	0-250	0.317	10107401	EPA625	Acceptable

Performance Summary

WP April 2003
Study Closing Date 05/16/2003

Product: Base Neutral Extractables **WP Lot Number: 35912** **APG+ Lot Number: 35965**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Dimethyl phthalate	WP	6135	98.4	118	0-181	0.614	10107401	EPA625	Acceptable
Di-n-butyl phthalate	WP	5925	73.6	85.2	15-120	0.343	10107401	EPA625	Acceptable
Di-n-octyl phthalate	WP	6200	47.4	73.4	12.8-108	0.811	10107401	EPA625	Acceptable
Fluoranthene	WP	6265	98.8	119	50.8-154	0.186	10107401	EPA625	Acceptable
Fluorene	WP	6270	38.0	47.4	20-61.5	0.405	10107401	EPA625	Acceptable
Hexachlorobenzene	WP	6275	167	189	82.8-243	0.149	10107401	EPA625	Acceptable
Hexachlorobutadiene	WP	4835	139	178	31.8-212	0.565	10107401	EPA625	Acceptable
Hexachlorocyclopentadiene	WP	6285	51.0	113	0-145	0.563	10107401	EPA625	Acceptable
Hexachloroethane	WP	4840	29.6	40.7	5.95-49.9	0.232	10107401	EPA625	Acceptable
Indeno(1,2,3-cd)pyrene	WP	6315	<5				10107401	EPA625	Acceptable
Isophorone	WP	6320	122	156	52.1-208	0.308	10107401	EPA625	Acceptable
Naphthalene	WP	5005	32.9	43	13.6-56.6	0.307	10107401	EPA625	Acceptable
Nitrobenzene (NB)	WP	5015	74.9	105	34.3-137	0.632	10107401	EPA625	Acceptable
N-nitrosodimethylamine	WP	6530	<5				10107401	EPA625	Acceptable
N-nitrosodi-n-propylamine	WP	6545	<5				10107401	EPA625	Acceptable
N-nitrosodiphenylamine	WP	6535	<5				10107401	EPA625	Acceptable
Phenanthrene	WP	6615	160	186	59.2-247	0.224	10107401	EPA625	Acceptable
Pyrene	WP	6665	29.4	35.5	11.4-52.1	0.354	10107401	EPA625	Acceptable

Product: BTEX **WP Lot Number: 35914** **APG+ Lot Number: 35967**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Benzene	WP	4375	53.9	58.1	42-74.7	0.806	10107207	EPA624	Acceptable
Ethylbenzene	WP	4765	40.1	41	27.4-53.5	0.0917	10107207	EPA624	Acceptable
m-Xylene / p-Xylene	WP	5240	191	218	182-244	2.14	10107207	EPA624	Check for Error
o-Xylene	WP	5250	126	140	111-162	1.28	10107207	EPA624	Acceptable
Toluene	WP	5140	<2				10107207	EPA624	Acceptable
Total Xylenes	WP	5260	318	357	206-474	0.492	10107207	EPA624	Acceptable

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Product: Demand WP Lot Number: 35892 APG+ Lot Number: 35946

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Biochemical Oxygen Demand	WP	1530	81.0	63	31.7-94.3	1.72	20027401	SM18thED5210B	Acceptable
Carbonaceous BOD	WP	1555	70.8	54.2	24.3-84.1	1.67	20027401	SM18thED5210B	Acceptable
Chemical Oxygen Demand	WP	1565	107	102	75.8-119	1.31	10076003	EPA410.1	Acceptable
Total Organic Carbon	WP	2040	41.6	40.2	33.5-46.4	0.744	10078407	EPA415.1	Acceptable

Product: Fluoride WP Lot Number: 35924 APG+ Lot Number: 35959

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Fluoride	WP	1730	1.64	1.75	1.51-1.97	1.32	10053006	EPA300.0	Acceptable

Product: Herbicides WP Lot Number: 35926 APG+ Lot Number: 35975

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
2,4,5-T	WP	8655	0.516	4.25	0.225-6.42	2.72	10105609	EPA615	Check for Error
2,4,5-TP (silvex)	WP	8650	1.07	3.46	0.106-4.97	1.81	10105609	EPA615	Acceptable
2,4-D	WP	8545	1.28	6.27	0-9.71	2.04	10105609	EPA615	Check for Error
Dicamba	WP	8595	0.755	5.58	0.426-8.16	2.74	10105609	EPA615	Check for Error
Dinoseb	WP	8620	3.42	3.69	0-3.49	2.89	10105609	EPA615	Check for Error

Product: Hexavalent Chromium WP Lot Number: 35910 APG+ Lot Number: 35961

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Chromium, Hexavalent	WP	1045	201	195	159-224	0.917	10162206	EPA7196	Acceptable

Performance Summary

WP April 2003
Study Closing Date 05/16/2003

Product: Minerals WP Lot Number: 35895-35896 APG+ Lot Number: 35950-35951

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Alkalinity as CaCO3	WP	1505	16.8	15.2	11.8-19.8	0.746	20003003	SM1819thED2320B	Acceptable
Calcium	WP	1035	36.3	35.7	31.7-40.6	0.0676	10014003	EPA200.7	Acceptable
Chloride	WP	1575	99.8	99.3	90.1-108	0.164	10053006	EPA300.0	Acceptable
Conductivity	WP	1610	554	556	507-595	0.203	10006209	EPA120.1	Acceptable
Magnesium	WP	1085	2.60	2.67	2.26-3.08	0.511	10014003	EPA200.7	Acceptable
Potassium	WP	1125	13.2	14.8	12.6-17	2.17	10014003	EPA200.7	Check for Error
Sodium	WP	1155	49.4	53.6	48.3-59	2.4	10014003	EPA200.7	Check for Error
Sulfate	WP	2000	79.5	77	63.7-88.2	0.88	10053006	EPA300.0	Acceptable
Total Dissolved Solids	WP	1705	300	309	227-379	0.119	10009004	EPA160.1	Acceptable
Total Hardness as CaCO3	WP	1755	101	100	89.8-111	0.287	20003401	SM1819ED2340B	Acceptable

Product: Nutrients WP Lot Number: 35893-35894 APG+ Lot Number: 35947-35948

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Ammonia Nitrogen as N	WP	1515	4.45	4.22	3.21-5.2	0.725	10064401	EPA350.3	Acceptable
Nitrate Nitrogen as N	WP	1810	6.84	6.72	5.3-8.01	0.421	10053006	EPA300.0	Acceptable
Orthophosphate as P	WP	1870	5.24	5.29	4.52-6.11	0.265	10053006	EPA300.0	Acceptable
Total Kjeldahl Nitrogen	WP	1795		20	14.6-24.7				
Total Phosphorus as P	WP	1910	2.81	2.61	1.98-3.08	1.53	10070801	EPA365.3	Acceptable

Product: Oil & Grease WP Lot Number: 35903 APG+ Lot Number: 35949

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Oil & Grease	WP	1860	25.4	23.7	14.3-28.7	1.62	10127409	EPA1664	Acceptable

Performance Summary

WP April 2003
Study Closing Date 05/16/2003

Product: PCB's in Oil **WP Lot Number: 35906-35907**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Aroclor 1016/1242 in Oil Sample 1	WP	8880	19.3	20.6	2.44-30.6	0.596	10103603	EPA608	Acceptable
Aroclor 1016/1242 in Oil Sample 2	WP	8880	<2				10103603	EPA608	Acceptable
Aroclor 1254 in Oil Sample 1	WP	8905	<2				10103603	EPA608	Acceptable
Aroclor 1254 in Oil Sample 2	WP	8905	<2				10103603	EPA608	Acceptable
Aroclor 1260 in Oil Sample 1	WP	8910	<2				10103603	EPA608	Acceptable
Aroclor 1260 in Oil Sample 2	WP	8910	38.9	45.8	8.28-64.4	0.278	10103603	EPA608	Acceptable

Product: PCB's in Water **WP Lot Number: 35904-35905** **APG+ Lot Number: 35971**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Aroclor 1016/1242 Sample 1	WP	8880	<1				10103603	EPA608	Acceptable
Aroclor 1016/1242 Sample 2	WP	8880	<1				10103603	EPA608	Acceptable
Aroclor 1232 Sample 1	WP	8890	<1				10103603	EPA608	Acceptable
Aroclor 1232 Sample 2	WP	8890	<1				10103603	EPA608	Acceptable
Aroclor 1248 Sample 1	WP	8900	<1				10103603	EPA608	Acceptable
Aroclor 1248 Sample 2	WP	8900	4.90	5.18	2.16-7.32	0.186	10103603	EPA608	Acceptable
Aroclor 1254 Sample 1	WP	8905	<1				10103603	EPA608	Acceptable
Aroclor 1254 Sample 2	WP	8905	<1				10103603	EPA608	Acceptable
Aroclor 1260 Sample 1	WP	8910	2.89	2.85	1.4-3.77	0.785	10103603	EPA608	Acceptable
Aroclor 1260 Sample 2	WP	8910	<1				10103603	EPA608	Acceptable

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Study Closing Date 05/16/2003

Product: Pesticides (NELAC) **WP Lot Number: 35929**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
alpha-BHC	WP	7110	5.02	6.51	2.41-9.01	0.627	10103603	EPA608	Acceptable
beta-BHC	WP	7115	7.71	10.2	3.81-14.7	0.845	10103603	EPA608	Acceptable
delta-BHC	WP	7105	9.77	11.6	3.27-15.4	0.218	10103603	EPA608	Acceptable
Endosulfan I	WP	7510	9.88	13.2	5.45-19.2	1.06	10103603	EPA608	Acceptable
Endosulfan II	WP	7515	31.0	39.6	7.18-64.2	0.495	10103603	EPA608	Acceptable
Endosulfan sulfate	WP	7520	5.71	6.05	1.84-9.37	0.0873	10103603	EPA608	Acceptable
Endrin	WP	7540	8.79	10.2	3.75-14.9	0.301	10103603	EPA608	Acceptable
Endrin aldehyde	WP	7530	8.72	10.2	3.34-14.9	0.218	10103603	EPA608	Acceptable
gamma-BHC	WP	7120	8.08	9.94	3.24-14.4	0.392	10103603	EPA608	Acceptable
Methoxychlor	WP	7810	3.31	3.2	0.994-5.35	0.193	10103603	EPA608	Acceptable

Product: Pesticides (WP) **WP Lot Number: 35928**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
4,4'-DDD	WP	7355	0.228	0.296	0.13-0.427	1.01	10103603	EPA608	Acceptable
4,4'-DDE	WP	7360	0.186	0.261	0.113-0.354	1.2	10103603	EPA608	Acceptable
4,4'-DDT	WP	7365	0.395	0.409	0.168-0.583	0.275	10103603	EPA608	Acceptable
Aldrin	WP	7025	0.433	0.537	0.134-0.728	0.0202	10103603	EPA608	Acceptable
Dieldrin	WP	7470	4.21	4.41	2.28-6.09	0.0472	10103603	EPA608	Acceptable
Heptachlor	WP	7685	2.61	3.07	0.808-4.28	0.121	10103603	EPA608	Acceptable
Heptachlor epoxide (beta)	WP	7690	2.13	2.3	1.17-2.96	0.235	10103603	EPA608	Acceptable

Product: pH **WP Lot Number: 35897** **APG+ Lot Number: 35952**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
pH	WP	1900	9.43	9.49	9.2-9.78	0.619	10008409	EPA150.1	Acceptable

Performance Summary

Product: Polynuclear Aromatic Hydrocarbons-PAH **WP Lot Number: 35913** **APG+ Lot Number: 35966**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Acenaphthene	WP	5500	1.89	2.52	1.2-2.64	0.124	10107401	EPA625	Acceptable
Acenaphthylene	WP	5505	<0.2				10107401	EPA625	Acceptable
Anthracene	WP	5555	0.37	0.502	0.211-0.588	0.478	10107401	EPA625	Acceptable
Benzo(a)anthracene	WP	5575	1.53	1.8	0-2.94	0.142	10107401	EPA625	Acceptable
Benzo(a)pyrene	WP	5580	0.41	0.498	0-0.956	0.156	10107401	EPA625	Acceptable
Benzo(b)fluoranthene	WP	5585	<0.2				10107401	EPA625	Acceptable
Benzo(g,h,i)perylene	WP	5590	<0.2				10107401	EPA625	Acceptable
Benzo(k)fluoranthene	WP	5600	<0.2				10107401	EPA625	Acceptable
Chrysene	WP	5855	0.50	0.552	0.135-0.834	0.137	10107401	EPA625	Acceptable
Dibenzo(a,h)anthracene	WP	5895	<0.2				10107401	EPA625	Acceptable
Fluoranthene	WP	6265	<0.2				10107401	EPA625	Acceptable
Fluorene	WP	6270	<0.2				10107401	EPA625	Acceptable
Indeno(1,2,3-cd)pyrene	WP	6315	<0.2				10107401	EPA625	Acceptable
Naphthalene	WP	5005	<0.2				10107401	EPA625	Acceptable
Phenanthrene	WP	6615	1.52	1.77	0.89-2.04	0.26	10107401	EPA625	Acceptable
Pyrene	WP	6665	<0.2				10107401	EPA625	Acceptable

Product: Solids **WP Lot Number: 83922**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Dissolved Solids	WP	1705	54.0	46.9	42.1-52.1	4.16	10009004	EPA160.1	**Not Acceptable**
Total Solids	WP	1950	109	107	95.2-119	0.51	10009800	EPA160.3	Acceptable
Total Suspended Solids	WP	1960	54.0	60.5	46.2-65	0.508	10009402	EPA160.2	Acceptable

Product: Total Chlordane **WP Lot Number: 35908** **APG+ Lot Number: 35974**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Chlordane (Total)	WP	7250	7.70	8.91	3.89-12.7	0.401	10103603	EPA608	Acceptable

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Product: Total Cyanide WP Lot Number: 35901 APG+ Lot Number: 35956

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Cyanide	WP	1645	0.617	0.641	0.444-0.825	0.268	10060409	EPA335.2	Acceptable

Product: Total Phenolics WP Lot Number: 35900 APG+ Lot Number: 35955

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Phenolics	WP	1905	0.214	0.221	0.0727-0.22	2.78	10079808	EPA420.2	Check for Error

Product: Total Residual Chlorine WP Lot Number: 35902 APG+ Lot Number: 35957

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Total Residual Chlorine	WP	1940	3.5	3.33	2.62-3.76	1.63	10059208	EPA330.4	Acceptable

Product: Toxaphene WP Lot Number: 35917 APG+ Lot Number: 35972

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Toxaphene	WP	8250	8.17	6.77	0.586-10.6	1.54	10103603	EPA608	Acceptable

Product: TPH Diesel (DRO) WP Lot Number: 35915 APG+ Lot Number: 35969

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
TPH Diesel (DRO)	WP	9369	0.23	0.474	0-0.764	0.536	10173203	EPA8015	Acceptable

Product: TPH Gasoline (GRO) WP Lot Number: 35916 APG+ Lot Number: 35968

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
TPH Gasoline (GRO)	WP	9408	2.21	2.56	1.08-3.1	0.357	10173203	EPA8015	Acceptable

Performance Summary

WP April 2003
Study Closing Date 05/16/2003

Product: Trace Metals **WP Lot Number: 35898-35899** **APG+ Lot Number: 35953-35954**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
Aluminum	WP	1000	2130	2150	1850-2440	0.102	10014003	EPA200.7	Acceptable
Antimony	WP	1005	746	802	568-963	0.289	10014003	EPA200.7	Acceptable
Arsenic	WP	1010	903	870	732-1020	0.591	10014003	EPA200.7	Acceptable
Barium	WP	1015	2220	2260	1920-2560	0.187	10014003	EPA200.7	Acceptable
Beryllium	WP	1020	525	563	479-636	1.22	10014003	EPA200.7	Acceptable
Boron	WP	1025	877	853	747-990	0.222	10156000	EPA6020	Acceptable
Cadmium	WP	1030	678	642	548-729	1.33	10014003	EPA200.7	Acceptable
Chromium	WP	1040	372	362	314-410	0.625	10014003	EPA200.7	Acceptable
Cobalt	WP	1050	1010	960	845-1080	1.3	10014003	EPA200.7	Acceptable
Copper	WP	1055	833	828	753-908	0.116	10014003	EPA200.7	Acceptable
Iron	WP	1070	2370	2270	2010-2560	0.883	10014003	EPA200.7	Acceptable
Lead	WP	1075	974	927	812-1040	1.31	10014003	EPA200.7	Acceptable
Manganese	WP	1090	3460	3340	3010-3710	0.847	10014003	EPA200.7	Acceptable
Mercury	WP	1095	12.3	14.6	10.9-18.2	1.9	10014809	EPA200.8	Acceptable
Molybdenum	WP	1100	81.0	81.5	67.5-94.4	0	10014003	EPA200.7	Acceptable
Nickel	WP	1105	1780	1590	1440-1770	3.09	10014003	EPA200.7	**Not Acceptable**
Selenium	WP	1140	1900	1900	1520-2200	0.351	10014003	EPA200.7	Acceptable
Silver	WP	1150	376	367	315-420	0.455	10014003	EPA200.7	Acceptable
Strontium	WP	1160	174	168	143-193	0.719	10014809	EPA 200.8	Acceptable
Thallium	WP	1165	478	457	368-531	1.06	10014003	EPA200.7	Acceptable
Tin	WP	1175	4500	4310	3390-5250	0.581	10014809	EPA200.8	Acceptable
Titanium	WP	1180	126	133	114-151	0.974	10014809	EPA200.8	Acceptable
Vanadium	WP	1185	7150	7120	6440-7830	0.0862	10014003	EPA200.7	Acceptable
Zinc	WP	1190	1270	1180	1040-1320	1.95	10014003	EPA200.7	Acceptable

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WP April 2003

Study Closing Date 05/16/2003

Product: Volatiles **WP Lot Number: 35909** **APG+ Lot Number: 35963**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code	Method Description	Evaluation
1,1,1-Trichloroethane	WP	5160	21.2	21.5	14.1-28.3	0	10107207	EPA624	Acceptable
1,1,2,2-Tetrachloroethane	WP	5110	153	167	98.6-233	0.58	10107207	EPA624	Acceptable
1,1,2-Trichloroethane	WP	5165	113	135	91.2-176	1.49	10107207	EPA624	Acceptable
1,1-Dichloroethane	WP	4630	<2				10107207	EPA624	Acceptable
1,1-Dichloroethene	WP	4640	52.7	66.8	36.6-102	1.52	10107207	EPA624	Acceptable
1,2-Dichloroethane	WP	4635	41.9	49	33.9-65.3	1.47	10107207	EPA624	Acceptable
1,2-Dichloropropane	WP	4655	11.6	11.7	9.09-14.4	0.114	10107207	EPA624	Acceptable
Bromodichloromethane	WP	4395	31.7	33.9	23.9-44.2	0.71	10107207	EPA624	Acceptable
Bromoform	WP	4400	18.9	21.2	13.1-29.2	0.818	10107207	EPA624	Acceptable
Bromomethane	WP	4950	<2				10107207	EPA624	Acceptable
Carbon tetrachloride	WP	4455	18.4	19	12-26.9	0.403	10107207	EPA624	Acceptable
Chlorobenzene	WP	4475	22.6	23.3	16.9-29.5	0.284	10107207	EPA624	Acceptable
Chloroethane	WP	4485	<2				10107207	EPA624	Acceptable
Chloroform	WP	4505	13.5	14	10-18.2	0.438	10107207	EPA624	Acceptable
Chloromethane	WP	4960	<2				10107207	EPA624	Acceptable
Dibromochloromethane	WP	4575	16.8	18	11.8-23.8	0.5	10107207	EPA624	Acceptable
Methylene chloride	WP	4975	10.1	11.3	7.18-16.3	1.12	10107207	EPA624	Acceptable
Tetrachloroethene	WP	5115	20.6	19.6	12.9-25.5	0.67	10107207	EPA624	Acceptable
trans-1,2-Dichloroethene	WP	4700	41.1	47.9	29.2-66.6	1.09	10107207	EPA624	Acceptable
trans-1,3-Dichloropropene	WP	4685	<2				10107207	EPA624	Acceptable
Trichloroethene	WP	5170	46.7	30.6	19.9-39.7	5.14	10107207	EPA624	**Not Acceptable**
Trichlorofluoromethane	WP	5175	<2				10107207	EPA624	Acceptable
Vinyl Chloride	WP	5235	<2				10107207	EPA624	Acceptable

WP Performance Summary

October 2003 WP

APG Customer Code: 3655

Analytical Management Lab
Suite B
15130 South Keeler
Olathe, KS 66062

APG

Introduction:

The Analytical Products Group, Inc. Proficiency Testing Program has been designed to evaluate your laboratory's performance against other participating laboratories on the same set of standards. This report has been specifically prepared for your organization to assist you in ensuring that your procedures are in-control. Therefore, in most cases sample levels are not designed to challenge the detection limits of the method.

The Analytical Products Group Proficiency Testing Program is certified to meet all of the following requirements:

- Accredited by NIST according to the National Voluntary Laboratory Accreditation Program (Handbook 150-19) for Chemical Calibrations: Providers of Proficiency Testing (NVLAB Code 200384-0)
- Analytical Products Group, Inc. Quality System is registered with DNVCertification to meet the ISO 9001 standard to design, manufacture, and distribute chemical reference materials and operate Proficiency Testing Programs (Certificate # 98-HOU-AQ-9513)
- Proficiency Testing Program is certified to meet all requirements as specified in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).
- Certified to meet ISO/REMCO Harmonized Protocol for Proficiency Testing of Analytical Laboratories
- Certified to meet ASTM E1301

Report Layout

This report contains the following sub-reports:

Introduction -This section is the general introduction to the Analytical Products Group, Inc. Proficiency Testing Program as well as all of the certifications that the organization maintains.

Terms & Definitions - Defines all of the terms used throughout the report. This is an excellent section to refer to first if you have any questions.

Laboratory Benchmark Report - This report presents an overall summary of the study as well as your performance relative to all other study participants.

Comment Report - Contains comments submitted to Analytical Products Group by the program participants or comments by Analytical Products Group to program participants concerning unusual characteristics of the data set or problems with standards which could influence the interpretation of the data. Please review this section thoroughly before examining your data.

Exception Report - This report is a summary of all of the analytes that resulted in an Evaluation of "Not Acceptable." The Exception Report is designed to very quickly identify for your laboratory the areas that may require your immediate attention.

Performance Evaluation Summary Report - Provides a summary of all of your data for each analyte. This summary is formatted similar to the US EPA DMR-QA Reports.

Detail Report- This report lists all the details of your data. Also included in this section are useful graphs indicating the APG Assigned Value, the EPA Mean, the EPA Acceptance Limits and your Reported Value. Another graph included in this section shows your Z score for the analyte of interest.

The goal of this report is to provide your laboratory with the information you need to monitor and improve your quality. If you have any questions or feedback, please give us a call (800-272-4442). Thank you for choosing Analytical Products Group as your proficiency testing provider.

Sincerely,

Thomas J. Coyner

Thomas J. Coyner
Proficiency Program Manager

Terms & Definitions

Average Acceptable Data - This value is the average percent of data that is within the EPA Acceptance Limits for this study. The Average Acceptable Data is reported for all laboratories as a whole as well as your laboratory as an individual. These values can be found in the Laboratory Benchmark Report.

Average Percentile for All Parameters - This is your laboratory's rank on each parameter and the related rank for that parameter. The average percentile is the average of this data across all parameters reported. It is a different measure of performance than percent acceptable data since it is affected by your relative performance on each parameter. The report provides an average for this measure for all laboratories reporting as well as your laboratory's performance.

Analyte - Name of the analyte of interest as found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) or NELAC Field of Testing List.

Analyte Code - This value is the code given to the analyte by the US EPA. These codes can be found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045). If no code is listed, then this analyte is not part of the US EPA program.

APG Assigned Value - This value is the calculated True Value of the standard based upon the actual composition of the standard.

Z-Score - This figure corresponds to how far your Reported Value was from the EPA Mean, reported in terms of EPA Standard Deviation units. As a point of reference, the US EPA Acceptance Limits are plus or minus three (3) EPA Standard Deviations from the EPA Mean.

APG Certified Value - This value is the result of the quality control testing performed on the sample by Analytical Products Group before the sample is shipped to the customer. Through this testing, the samples are traced against a NIST Standard Reference Material where available or an external standard if a NIST Standard Reference Material is not available. The reported uncertainty of this value is expressed as two times the standard deviation (s) divided by the square root of the number of samples (n) tested (i.e. 2s/vn). This quality control testing meets all of the requirements as specified by the NIST National Voluntary Laboratory Accreditation Program (NVLAP) Handbook 150-19 (Chemical Calibration: Providers of Proficiency Testing).

EPA Acceptance Range - This range is calculated based upon the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045). For the Water Pollution Program (WP), EPA Acceptance Limits are defined as ± three (3) EPA Standard Deviations from the EPA Mean.

EPA Mean - If the analyte is included in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then this value is the Mean Value calculated per the Criteria Document. If the analyte is not included in the Criteria Document, then this value is the Study Mean.

Study Standard Deviation - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Standard Deviation is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Your Ranking - This is your ranking based on your Z Score for the analyte of interest among all possible rankings. It is important to note that several laboratories can achieve the same rank.

Evaluation - This value is determined by the EPA Acceptance Limits and the Reported Value. Evaluation may have one of following values:

Value	Description
Acceptable	The Reported Value was within the EPA Acceptable Limits or a less than (<) value was reported AND zero (0) was included in the EPA Acceptance Limit Range AND the numeric part of the Reported Value was less than the upper limit of the EPA Acceptance Limit.
Not Acceptable	The Reported Value is outside of the EPA Acceptance Limits or a less than (<) value was reported AND zero (0) is not included in the EPA Acceptance Limits range or the numeric part of the Reported Value was greater than the upper limit of the EPA Acceptance Limit or a greater than value (>) was reported.
Check for Error	The Reported Value is between two (2) and three (3) EPA Standard Deviations from the EPA Mean.
Unusable	The Reported Value was a text value which could not be evaluated.
Blank	No data was reported for this analyte or the analyte was not spiked.

Inter-Laboratory Statistics - This data is a summary of all of the data from all study participants for the analyte of interest. Inter-Laboratory Statistics are only reported if a minimum number of laboratories analyzed and reported data for the analyte of interest.

Program - This value indicates which program the analyte of interest is a part. For Water Pollution Studies (WP), the only values that Program will have are 'WP' or 'APG+'. If the analyte and range are consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then the Program value will have a value of 'WP' and if this is not the case then the Program value will be 'APG+', indicating that the analyte and/or range was specified by Analytical Products Group.

Rank on Percent Acceptable Data - This is your laboratories rank out of all possible ranks in the current study based on percent acceptable data. It is important to note that several laboratories can achieve the same rank.

Rank on Average Percentile Data - This value is your laboratories rank out of all possible ranks based on Your Ranking converted to a percentile. It is important to note that several laboratories can achieve the same rank.

Reported Value - This value is the result that was reported by your organization for the particular analyte. Every effort has been made to ensure correct data entry. Should you find a data entry error, please notify Analytical Products Group, Inc. and we will correct the error and re-issue the report. If the Reported Value is reported as a less than (<) than figure, then the only way that the Evaluation will result in an "Acceptable" value is if the numeric part of the Reported Value (i.e. 8 if the Report Value were <8) is less then the upper limit of the EPA Acceptance Limit AND zero (0) is included in the EPA Acceptance Limit. Any other non-numeric value (i.e. BDL, ND, >10, etc.) cannot be evaluated and will return an Evaluation result of "Unusable." This method of evaluation is consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).

Solids Standards - Solids standards are individually prepared, therefore, each standard has a unique APG Assigned Value.

Terms & Definitions (continued)

Laboratory Pass Percentage - This is the percentage of study participants that reported values within the EPA Acceptance Limits for the analyte of interest.

Number of Acceptable Data Points - This value is the number of data points reported by your laboratory that were within the EPA Acceptance Limits.

Weighted Rank on Percent Acceptable Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percent Acceptable Data is the weighted rank of your laboratory based on Percent Acceptable Data and the number of analytes reported.

Weight Rank on Percentile Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percentile Data is the weighted rank of your laboratory based on Rank on Average Percentile Data and the number of analytes reported.

Study Mean - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Mean is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Laboratory Benchmark Report

Customer Code: 3655

October 2003 WP

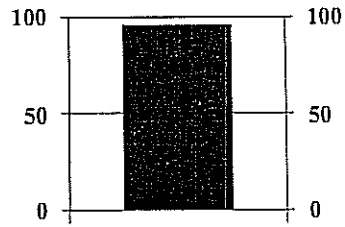
Summary of Ranking Data

Summary of National Ranking Data

Total Number of Laboratories Reporting:	278
Average Percent Acceptable Data:	96.15%
Average Percentile for all Parameters:	54.00%

National Avg % Acceptable

96.2 %

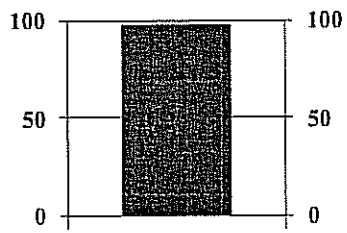


Summary of Laboratory Ranking:

Total Data Points Reported:	216
Number of Acceptable Data Points:	212
Percent Acceptable Data:	98.15%
Average Percentile for all Parameters:	56.88%

Laboratory Avg % Acceptable

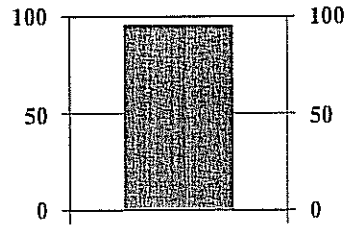
98.15%



Rank on Percent Acceptable Data:	20 of 86
Rank on Average Percentile Data:	116 of 249
Weighted Rank on Percent Acceptable Data:	14 of 124
Weighted Rank on Average Percentile Data:	11 of 262

Weighted Rank Avg Percentile

95.8 %



There are no comments to report.

Exception Report

APG Customer	3655	Labratory Address	Nelac Point Of Contact	WP October 2003 WP #3010	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003

Product: Demand — **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Organic Carbon	WP	2040	7.76	14.2	11.7-16.7	7.73	10078407 EPA415.1	402010 IR	Not Acceptable

Product: Nutrients — **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Ammonia Nitrogen as N	WP	1515	22.9	17.8	13.9-21.6	4.0625	10064207 EPA350.3	352010 POT	Not Acceptable

Product: Polynuclear Aromatic Hydrocarbons-PAH — **Exception Reporting**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Chrysene	WP	5855	0.23			4.0625	10107401 EPA625	151010 GC-MS	Not Acceptable
Pyrene	WP	6665	0.62	0.381	0.121-0.506	4.766355	10107401 EPA625	151010 GC-MS	Not Acceptable

Performance Summary

APG Customer	3655	Labratory Address	Nelac Point Of Contact	WP October 2003 WP #3010	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003

Product: Acid Extractables WP Lot Number: 37256 APG+ Lot Number: 37257

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
2,4,5-Trichlorophenol	WP	6835	95.5	124	46.3-158	0.351	10107401 EPA625	151010 GC-MS	Acceptable
2,4,6-Trichlorophenol	WP	6840	96.3	125	40.8-166	0.322	10107401 EPA625	151010 GC-MS	Acceptable
2,4-Dichlorophenol	WP	6000	90.9	132	47.6-159	0.654	10107401 EPA625	151010 GC-MS	Acceptable
2,4-Dimethylphenol	WP	6130	76.9	109	23.1-143	0.31	10107401 EPA625	151010 GC-MS	Acceptable
2,4-Dinitrophenol	WP	6175	83.0	124	0-163	0.15602	10107401 EPA625	151010 GC-MS	Acceptable
2-Chlorophenol	WP	5800	44.1	67.4	20.6-85.4	0.824	10107401 EPA625	151010 GC-MS	Acceptable
2-Methyl-4,6-Dinitrophenol	WP	6360	124	158	16.9-231	0	10107401 EPA625	151010 GC-MS	Acceptable
2-Nitrophenol	WP	6490	55.9	76.7	23.3-100	0.465	10107401 EPA625	151010 GC-MS	Acceptable
4-Chloro-3-methylphenol	WP	5700	153	188	74.5-241	0.18	10107401 EPA625	151010 GC-MS	Acceptable
4-Nitrophenol	WP	6500	63.4	88.7	0-123	0.56504	10107401 EPA625	151010 GC-MS	Acceptable
Pentachlorophenol	WP	6605	29.8	42.8	7.31-59.1	0.394	10107401 EPA625	151010 GC-MS	Acceptable
Phenol	WP	6625	52.7	91.5	0-124	0.04545	10107401 EPA625	151010 GC-MS	Acceptable

Produ Aluminum High Level WP Lot Number: 37227 APG+ Lot Number: 37228

	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Level	WP	1000	0.708	0.751	0.647-0.832	1.04	10014003 EPA200.7	102015 ICP-AES	Acceptable

Performance Summary

APG Customer 3655	Labratory Address	Nelac Point Of Contact		WP October 2003 WP #3010
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003
Primary AA E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003

Product: Aromatic Hydrocarbons **WP Lot Number: 37439** **APG+ Lot Number: 37440**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
1,2-Dichlorobenzene	WP	4610	33.7	36.2	25.4-46	0.585	10107207 EPA624	151010 GC-MS	Acceptable
1,3-Dichlorobenzene	WP	4615	14.7	14.9	10.2-19	0.06849	10107207 EPA624	151010 GC-MS	Acceptable
1,4-Dichlorobenzene	WP	4620	45.8	48.1	33.1-62	0.353	10107207 EPA624	151010 GC-MS	Acceptable
Benzene	WP	4375	26.3	24.9	17.7-32.4	0.53278	10107207 EPA624	151010 GC-MS	Acceptable
Ethylbenzene	WP	4765	66.3	60.2	40.1-78.8	1.06976	10107207 EPA624	151010 GC-MS	Acceptable
m-Xylene / p-Xylene	WP	5240	74.3	74.6	55.9-98.9	0.432	10107207 EPA624	151010 GC-MS	Acceptable
o-Xylene	WP	5250	196	208	148-275	0.758	10107207 EPA624	151010 GC-MS	Acceptable
Toluene	WP	5140	26.7	25.9	18.4-32.7	0.50420	10107207 EPA624	151010 GC-MS	Acceptable
Total Xylenes	WP	5260	271	283	163-377	0.02808	10107207 EPA624	151010 GC-MS	Acceptable

Performance Summary

APG Customer	3655	Labratory Address	Nelac Point Of Contact		WP October 2003 WP #3010
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003

Product: Base Neutral Extractables **WP Lot Number: 37299** **APG+ Lot Number: 37300**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
1,2,4-Trichlorobenzene	WP	5155	<10			0.02808	10107401 EPA625	151010 GC-MS	Acceptable
1,2-Dichlorobenzene	WP	4610	21.8	37.7	12.3-39.4	0.907	10107401 EPA625	151010 GC-MS	Acceptable
1,3-Dichlorobenzene	WP	4615	34.4	61.2	17.5-60.1	0.619	10107401 EPA625	151010 GC-MS	Acceptable
1,4-Dichlorobenzene	WP	4620	15.9	27.4	7.84-28.9	0.712	10107401 EPA625	151010 GC-MS	Acceptable
2,4-Dinitrotoluene (2,4-DNT)	WP	6185	167	187	81-250	0.07117	10107401 EPA625	151010 GC-MS	Acceptable
2,6-Dinitrotoluene (2,6-DNT)	WP	6190	116	135	51.6-179	0.04739	10107401 EPA625	151010 GC-MS	Acceptable
2-Chloronaphthalene	WP	5795	27.2	38.6	14.8-47.9	0.762	10107401 EPA625	151010 GC-MS	Acceptable
3,3'-Dichlorobenzidine	WP	5945	<10			0.762	10107401 EPA625	151010 GC-MS	Acceptable
4-Bromophenyl phenyl ether	WP	5660	<10			0.762	10107401 EPA625	151010 GC-MS	Acceptable
4-Chlorophenyl phenyl ether	WP	5825	<10			0.762	10107401 EPA625	151010 GC-MS	Acceptable
Acenaphthene	WP	5500	27.2	35	14.1-44.8	0.451	10107401 EPA625	151010 GC-MS	Acceptable
Acenaphthylene	WP	5505	46.6	54.4	21.1-67.2	0.3125	10107401 EPA625	151010 GC-MS	Acceptable
Anthracene	WP	5555	95.4	111	38.9-143	0.24712	10107401 EPA625	151010 GC-MS	Acceptable
Benzidine	WP	5595	<10			0.24712	10107401 EPA625	151010 GC-MS	Acceptable

Performance Summary

APG Customer	3655	Labratory Address	Nelac Point Of Contact		WP October 2003 WP #3010
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003

Product: Base Neutral Extractables **WP Lot Number: 37299** **APG+ Lot Number: 37300**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Benzo(a)anthracene	WP	5575	33.5	38.7	22-45.3	0.0514	10107401 EPA625	151010 GC-MS	Acceptable
Benzo(a)pyrene	WP	5580	32.4	41.2	7.65-54.4	0.17948	10107401 EPA625	151010 GC-MS	Acceptable
Benzo(b)fluoranthene	WP	5585	<10			0.17948	10107401 EPA625	151010 GC-MS	Acceptable
Benzo(g,h,i)perylene	WP	5590	<10			0.17948	10107401 EPA625	151010 GC-MS	Acceptable
Benzo(k)fluoranthene	WP	5600	<10			0.17948	10107401 EPA625	151010 GC-MS	Acceptable
Bis(2-Chloroethoxy)methane	WP	5760	<10			0.17948	10107401 EPA625	151010 GC-MS	Acceptable
Bis(2-Chloroethyl)ether	WP	5765	<10			0.17948	10107401 EPA625	151010 GC-MS	Acceptable
Bis(2-Chloroisopropyl)ether	WP	5780	<10			0.17948	10107401 EPA625	151010 GC-MS	Acceptable
Bis(2-Ethylhexyl)phthalate	WP	6255	56.2	70.6	17.4-103	0.268	10107401 EPA625	151010 GC-MS	Acceptable
Butyl benzyl phthalate	WP	5670	45.6	51.5	0-70.3	0.90677	10107401 EPA625	151010 GC-MS	Acceptable
Chrysene	WP	5855	90.4	105	39.4-138	0.09090	10107401 EPA625	151010 GC-MS	Acceptable
Dibenzo(a,h)anthracene	WP	5895	<10			0.09090	10107401 EPA625	151010 GC-MS	Acceptable
Diethyl phthalate	WP	6070	139	164	0-251	0.47191	10107401 EPA625	151010 GC-MS	Acceptable
Dimethyl phthalate	WP	6135	66.2	76.5	0-117	0.55288	10107401 EPA625	151010 GC-MS	Acceptable

Performance Summary

APG Customer 3655	Labratory Address	Nelac Point Of Contact	WP October 2003 WP #3010		
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003	
Primary AA E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003	
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003	

Product: Base Neutral Extractables **WP Lot Number: 37299** **APG+ Lot Number: 37300**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Di-n-butyl phthalate	WP	5925	143	160	16.6-223	0.66860	10107401 EPA625	151010 GC-MS	Acceptable
Di-n-octyl phthalate	WP	6200	26.2	32.8	8.98-50	0.482	10107401 EPA625	151010 GC-MS	Acceptable
Fluoranthene	WP	6265	152	180	75.1-233	0.076	10107401 EPA625	151010 GC-MS	Acceptable
Fluorene	WP	6270	83.8	104	43.6-133	0.309	10107401 EPA625	151010 GC-MS	Acceptable
Hexachlorobenzene	WP	6275	55.4	64.3	28.1-83.2	0.0218	10107401 EPA625	151010 GC-MS	Acceptable
Hexachlorobutadiene	WP	4835	36.1	63.6	9.81-77.6	0.673	10107401 EPA625	151010 GC-MS	Acceptable
Hexachlorocyclopentadiene	WP	6285	68.3	122	0-156	0.0954	10107401 EPA625	151010 GC-MS	Acceptable
Hexachloroethane	WP	4840	25.8	49.1	6.41-60.7	0.862	10107401 EPA625	151010 GC-MS	Acceptable
Indeno(1,2,3-cd)pyrene	WP	6315	<10			0.862	10107401 EPA625	151010 GC-MS	Acceptable
Isophorone	WP	6320	53.6	69.6	25.4-91.9	0.459	10107401 EPA625	151010 GC-MS	Acceptable
Naphthalene	WP	5005	108	165	39.1-215	0.648	10107401 EPA625	151010 GC-MS	Acceptable
Nitrobenzene (NB)	WP	5015	68.3	101	33-132	0.855	10107401 EPA625	151010 GC-MS	Acceptable
N-nitrosodimethylamine	WP	6530	<10			0.855	10107401 EPA625	151010 GC-MS	Acceptable
N-nitrosodi-n-propylamine	WP	6545	<10			0.855	10107401 EPA625	151010 GC-MS	Acceptable

Performance Summary

APG Customer	3655	Labratory Address	Nelac Point Of Contact	WP October 2003 WP #3010	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003

Product: Base Neutral Extractables WP Lot Number: 37299 APG+ Lot Number: 37300

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
N-nitrosodiphenylamine	WP	6535	<10			0.855	10107401 EPA625	151010 GC-MS	Acceptable
Phenanthrene	WP	6615	145	176	56.7-233	0	10107401 EPA625	151010 GC-MS	Acceptable
Pyrene	WP	6665	117	140	46.6-188	0	10107401 EPA625	151010 GC-MS	Acceptable

Product: BTEX WP Lot Number: 37306 APG+ Lot Number: 37307

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Benzene	WP	4375	<0.5			0	10107207 EPA624	151010 GC-MS	Acceptable
Ethylbenzene	WP	4765	37.6	36.9	24.7-48.2	0.30690	10107207 EPA624	151010 GC-MS	Acceptable
m-Xylene / p-Xylene	WP	5240	30.4	26.9	18.4-37.6	0.75	10107207 EPA624	151010 GC-MS	Acceptable
o-Xylene	WP	5250	176	180	132-232	0.359	10107207 EPA624	151010 GC-MS	Acceptable
Toluene	WP	5140	35.7	36.2	25.9-45.4	0.03076	10107207 EPA624	151010 GC-MS	Acceptable
Total Xylenes	WP	5260	206	207	119-276	0.30534	10107207 EPA624	151010 GC-MS	Acceptable

Performance Summary

APG Customer	3655	Labratory Address	Nelac Point Of Contact	WP October 2003 WP #3010	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003

Product: Demand WP Lot Number: 37217 APG+ Lot Number: 36774

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Biochemical Oxygen Demand	WP	1530	25.3	22.3	11-33.6	0.79330	10075602 EPA405.1	352010 POT	Acceptable
Carbonaceous BOD	WP	1555	23.9	19.3	8.62-30	1.29210	20027401 SM18thED5210B	352010 POT	Acceptable
Chemical Oxygen Demand	WP	1565	36.3	35.9	22.2-47.1	0.41062	10077200 EPA410.4	401010 COLOR	Acceptable
Total Organic Carbon	WP	2040	7.76	14.2	11.7-16.7	7.73	10078407 EPA415.1	402010 IR	Not Acceptable

Product: Fluoride WP Lot Number: 37331 APG+ Lot Number: 36539

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Fluoride	WP	1730	2.27	2.46	2.14-2.75	1.76	10053006 EPA300.0	251510 IC	Acceptable

Product: Herbicides WP Lot Number: 37258 APG+ Lot Number: 37259

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
2,4,5-T	WP	8655	1.62	8.6	0.956-12.5	2.66	10105609 EPA615	152010 GC-ECD	Check for Error
2,4,5-TP (silvex)	WP	8650	0.833	2.4	0.0271-3.64	1.67	10105609 EPA615	152010 GC-ECD	Acceptable
2,4-D	WP	8545	1.06	5.27	0-8.33	2.03	10105609 EPA615	152010 GC-ECD	Check for Error
Dicamba	WP	8595	1.06	7.8	0.685-11.3	2.8	10105609 EPA615	152010 GC-ECD	Check for Error
Dinoseb	WP	8620	4.73	6.79	0-13	0.29	10105609 EPA615	152010 GC-ECD	Acceptable

Performance Summary

APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Labratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP October 2003 WP #3010
Study Print Date 11/26/2003
Study Shipment Date 10/01/2003
Study Closing Date 11/14/2003

Product: Hexavalent Chromium WP Lot Number: 37230 APG+ Lot Number: 37231

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Chromium, Hexavalent	WP	1045	240	223	182-256	1.68	10162206 EPA7196	401010 COLOR	Acceptable

Product: Minerals WP Lot Number: 37332-37333 APG+ Lot Number: 37334-37335

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Alkalinity as CaCO3	WP	1505	53.5	51.3	45.5-57.5	0.99502	10054805 EPA310.1	651010 TITR	Acceptable
Calcium	WP	1035	26.1	26.4	23.4-30.1	0.619	10014003 EPA200.7	102015 ICP-AES	Acceptable
Chloride	WP	1575	160	164	150-178	0.844	10057406 EPA325.3	651010 TITR	Acceptable
Chloride	WP	1575	162	164	150-178	0.422	10053006 EPA300.0	251510 IC	Acceptable
Conductivity	WP	1610	713	730	659-777	0.255	10006209 EPA120.1	351010 COND	Acceptable
Magnesium	WP	1085	19.7	20.5	17.9-23	0.826	10014003 EPA200.7	102015 ICP-AES	Acceptable
Potassium	WP	1125	36.1	39	33.8-44.4	1.69	10014003 EPA200.7	102015 ICP-AES	Acceptable
Sodium	WP	1155	46.5	49.4	44.5-54.5	1.8	10014003 EPA200.7	102015 ICP-AES	Acceptable
Sulfate	WP	2000	22.6	24.4	19.3-28.9	0.932	10073606 EPA375.4	401010 COLOR	Acceptable
Sulfate	WP	2000	24.1	24.4	19.3-28.9	0	10053006 EPA300.0	251510 IC	Acceptable
Total Dissolved Solids	WP	1705	308	432	294-480	2.56	10009208 EPA160.1	201010 GRAV	Check for Error
Total Hardness as CaCO3	WP	1755	146	151	137-166	1.04	20003401 SM1819ED2340B	102015 ICP-AES	Acceptable

Performance Summary

APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Labratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP October 2003 WP #3010
Study Print Date 11/26/2003
Study Shipment Date 10/01/2003
Study Closing Date 11/14/2003

Product: Nutrients WP Lot Number: 37241-37242 APG+ Lot Number: 36710-36711

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Ammonia Nitrogen as N	WP	1515	22.9	17.8	13.9-21.6	4.0625	10064207 EPA350.3	352010 POT	Not Acceptable
Nitrate Nitrogen as N	WP	1810	2.70	3.07	2.4-3.68	1.59	10068005 EPA353.3	401010 COLOR	Acceptable
Nitrate Nitrogen as N	WP	1810	2.69	3.07	2.4-3.68	1.64	10053006 EPA300.0	251510 IC	Acceptable
Orthophosphate as P	WP	1870	4.54	4.26	3.64-4.92	1.21495	10070607 EPA365.3	401010 COLOR	Acceptable
Orthophosphate as P	WP	1870	4.11	4.26	3.64-4.92	0.794	10053006 EPA300.0	251510 IC	Acceptable
Total Kjeldahl Nitrogen	WP	1795		13.4	9.67-16.7	0.794			Not Reported
Total Phosphorus as P	WP	1910	3.22	3.49	2.65-4.11	0.661	10070607 EPA365.3	401010 COLOR	Acceptable

Product: Oil & Grease WP Lot Number: 37394 APG+ Lot Number: 37395

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Oil & Grease	WP	1860	32.3	39.2	25.8-44.9	0.943	10127409 EPA1664	201010 GRAV	Acceptable

Performance Summary

APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Labratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP October 2003 WP #3010
Study Print Date 11/26/2003
Study Shipment Date 10/01/2003
Study Closing Date 11/14/2003

Product: PCB's in Oil WP Lot Number: 37385-37386

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aroclor 1016/1242 in Oil Sample 1	WP	8880	<1			0.943	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1016/1242 in Oil Sample 2	WP	8880	25.5	42	7.8-56.1	0.807	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1254 in Oil Sample 1	WP	8905	18.1	21.4	1.41-33.7	0.11152	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1254 in Oil Sample 2	WP	8905	<1			0.11152	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1260 in Oil Sample 1	WP	8910	<1			0.11152	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1260 in Oil Sample 2	WP	8910	<1			0.11152	10103603 EPA608	152010 GC-ECD	Acceptable

Performance Summary

APG Customer 3655	Labratory Address	Nelac Point Of Contact		WP October 2003 WP #3010
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003
Primary AA E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003

Product: PCB's in Water **WP Lot Number: 37401-37402** **APG+ Lot Number: 37403**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aroclor 1016/1242 Sample 1	WP	8880	<0.1			0.11152	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1016/1242 Sample 2	WP	8880	<0.1			0.11152	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1232 Sample 1	WP	8890	<0.1			0.11152	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1232 Sample 2	WP	8890	<0.1			0.11152	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1248 Sample 1	WP	8900	2.17	2.41	0.984-3.33	0.02557	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1248 Sample 2	WP	8900	<0.1			0.02557	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1254 Sample 1	WP	8905	<0.1			0.02557	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1254 Sample 2	WP	8905	<0.1			0.02557	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1260 Sample 1	WP	8910	<0.1			0.02557	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1260 Sample 2	WP	8910	1.90	2.03	0.885-2.72	0.32679	10103603 EPA608	152010 GC-ECD	Acceptable

Performance Summary

APG Customer	3655	Labratory Address	Nelac Point Of Contact	WP October 2003 WP #3010	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003
APG NIST/NVLAP 200384-0		Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003

Product: Pesticides (NELAC) **WP Lot Number: 37460**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
alpha-BHC	WP	7110	5.69	6.57	2.43-9.1	0.0631	10103603 EPA608	152010 GC-ECD	Acceptable
beta-BHC	WP	7115	7.72	7.82	2.87-11.3	0.44680	10103603 EPA608	152010 GC-ECD	Acceptable
delta-BHC	WP	7105	10.9	10.5	3.06-13.7	1.41011	10103603 EPA608	152010 GC-ECD	Acceptable
Endosulfan I	WP	7510	14.8	14.2	5.87-20.7	0.60728	10103603 EPA608	152010 GC-ECD	Acceptable
Endosulfan II	WP	7515	25.3	24.7	4.98-39.5	0.53913	10103603 EPA608	152010 GC-ECD	Acceptable
Endosulfan sulfate	WP	7520	24.4	22.9	3.64-37.8	0.65026	10103603 EPA608	152010 GC-ECD	Acceptable
Endrin	WP	7540	12.3	12.5	4.51-18.5	0.34334	10103603 EPA608	152010 GC-ECD	Acceptable
Endrin aldehyde	WP	7530	13.3	14.4	4.71-21.1	0.14652	10103603 EPA608	152010 GC-ECD	Acceptable
gamma-BHC	WP	7120	18.5	19.1	6-27.8	0.44077	10103603 EPA608	152010 GC-ECD	Acceptable
Methoxychlor	WP	7810	14.1	15.8	6.61-22.3	0.153	10103603 EPA608	152010 GC-ECD	Acceptable

Performance Summary

APG Customer	3655	Labratory Address	Nelac Point Of Contact	WP October 2003 WP #3010	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003

Product: Pesticides (WP) **WP Lot Number: 37459**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
4,4'-DDD	WP	7355	0.420	0.489	0.225-0.695	0.511	10103603 EPA608	152010 GC-ECD	Acceptable
4,4'-DDE	WP	7360	0.602	0.724	0.334-0.986	0.532	10103603 EPA608	152010 GC-ECD	Acceptable
4,4'-DDT	WP	7365	3.77	3.97	1.81-5.38	0.30303	10103603 EPA608	152010 GC-ECD	Acceptable
Aldrin	WP	7025	0.256	0.477	0.119-0.647	1.44	10103603 EPA608	152010 GC-ECD	Acceptable
Dieldrin	WP	7470	2.79	3.11	1.6-4.29	0.357	10103603 EPA608	152010 GC-ECD	Acceptable
Heptachlor	WP	7685	0.223	0.382	0.108-0.528	1.36	10103603 EPA608	152010 GC-ECD	Acceptable
Heptachlor epoxide (beta)	WP	7690	1.50	1.79	0.909-2.3	0.474	10103603 EPA608	152010 GC-ECD	Acceptable

Product: pH **WP Lot Number: 37796** **APG+ Lot Number: 37797**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
pH	WP	1900	7.97	8.03	7.8-8.26	0.792	10008409 EPA150.1	352010 POT	Acceptable

Performance Summary

APG Customer	3655	Labratory Address	Nelac Point Of Contact		WP October 2003 WP #3010
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003
APG NIST/NVLAP 200384-0		Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003

Product: Polynuclear Aromatic Hydrocarbons-PAH **WP Lot Number:** 37301 **APG+ Lot Number:** 37302

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Acenaphthene	WP	5500	11.5	18.2	5.59-21.3	0.728	10107401 EPA625	151010 GC-MS	Acceptable
Acenaphthylene	WP	5505	10.2	13.3	3.82-15.3	0.32812	10107401 EPA625	151010 GC-MS	Acceptable
Anthracene	WP	5555	0.84	0.938	0.295-1.23	0.49358	10107401 EPA625	151010 GC-MS	Acceptable
Benzo(a)anthracene	WP	5575	<0.2			0.49358	10107401 EPA625	151010 GC-MS	Acceptable
Benzo(a)pyrene	WP	5580	<0.2			0.49358	10107401 EPA625	151010 GC-MS	Acceptable
Benzo(b)fluoranthene	WP	5585	<0.2			0.49358	10107401 EPA625	151010 GC-MS	Acceptable
Benzo(g,h,i)perylene	WP	5590	<0.2			0.49358	10107401 EPA625	151010 GC-MS	Acceptable
Benzo(k)fluoranthene	WP	5600	<0.2			0.49358	10107401 EPA625	151010 GC-MS	Acceptable
Chrysene	WP	5855	0.23			0.49358	10107401 EPA625	151010 GC-MS	Not Acceptable
Dibenzo(a,h)anthracene	WP	5895	<0.2			0.49358	10107401 EPA625	151010 GC-MS	Acceptable
Fluoranthene	WP	6265	1.16	1.22	0.5-1.45	1.17088	10107401 EPA625	151010 GC-MS	Acceptable
Fluorene	WP	6270	2.71	4.07	1.51-4.54	0.614	10107401 EPA625	151010 GC-MS	Acceptable
Indeno(1,2,3-cd)pyrene	WP	6315	<0.2			0.614	10107401 EPA625	151010 GC-MS	Acceptable
Naphthalene	WP	5005	7.75	12.9	3.25-14.4	0.573	10107401 EPA625	151010 GC-MS	Acceptable

Performance Summary

APG Customer	3655	Labratory Address	Nelac Point Of Contact	WP October 2003 WP #3010	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003

Product: Polynuclear Aromatic Hydrocarbons-PAH WP Lot Number: 37301 APG+ Lot Number: 37302

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Phenanthrene	WP	6615	1.55	1.73	0.593-2.17	0.64393	10107401 EPA625	151010 GC-MS	Acceptable
Pyrene	WP	6665	0.62	0.381	0.121-0.506	4.76635	10107401 EPA625	151010 GC-MS	Not Acceptable

Product: Solids WP Lot Number: 87652

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Dissolved Solids	WP	1705	570	618	473-761	0.981	10009208 EPA160.1	201010 GRAV	Acceptable
Total Solids	WP	1950	661	701	530-892	0.829	10009800 EPA160.3	201010 GRAV	Acceptable
Total Suspended Solids	WP	1960	80	83	64.2-89.6	0.73286	10009606 EPA160.2	201010 GRAV	Acceptable

Product: Total Chlordane WP Lot Number: 37264 APG+ Lot Number: 37265

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Chlordane (Total)	WP	7250	2.09	2.41	1.06-3.42	0.381	10103603 EPA608	152010 GC-ECD	Acceptable

Product: Total Cyanide WP Lot Number: 37351 APG+ Lot Number: 37352

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Cyanide	WP	1645	0.732	0.666	0.461-0.857	1.10773	10060409 EPA335.2	401010 COLOR	Acceptable

Product: Total Phenolics WP Lot Number: 37347 APG+ Lot Number: 37348

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Phenolics	WP	1905	0.791	1.61	0.586-1.55	1.74	10079604 EPA420.2	401010 COLOR	Acceptable

Performance Summary

APG Customer	3655	Labratory Address	Nelac Point Of Contact	WP October 2003 WP #3010	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003
APG NIST/NVLAP 200384-0		Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003

Product: Total Residual Chlorine — **WP Lot Number: 37371** — **APG+ Lot Number: 37372**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code / Method Desc.	Technology Code / Technology Desc.	Evaluation
Total Residual Chlorine	WP	1940	1.86	1.72	1.33-1.99	1.81818	10059208 / EPA330.4	651010 / TITR	Acceptable

Product: Toxaphene — **WP Lot Number: 37266** — **APG+ Lot Number: 37267**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code / Method Desc.	Technology Code / Technology Desc.	Evaluation
Toxaphene	WP	8250	3.82	4.91	0.93-8.96	0.836	10103603 / EPA608	152010 / GC-ECD	Acceptable

Product: TPH Diesel (DRO) — **WP Lot Number: 37415** — **APG+ Lot Number: 37416**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code / Method Desc.	Technology Code / Technology Desc.	Evaluation
TPH Diesel (DRO)	WP	9369	2.40	4.02	1.4-5.26	1.45	10173203 / EPA8015	152510 / GC-FID	Acceptable

Product: TPH Gasoline (GRO) — **WP Lot Number: 37417** — **APG+ Lot Number: 37418**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code / Method Desc.	Technology Code / Technology Desc.	Evaluation
TPH Gasoline (GRO)	WP	9408	0.707	0.852	0.276-1.36	0.611	10173203 / EPA8015	152510 / GC-FID	Acceptable

Performance Summary

APG Customer	3655	Labratory Address	Nelac Point Of Contact		WP October 2003 WP #3010
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003

Product: Trace Metals **WP Lot Number: 37208-37209** **APG+ Lot Number: 36544-36545**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aluminum	WP	1000	278	280	227-335	0.167	10014003 EPA200.7	102015 ICP-AES	Acceptable
Antimony	WP	1005	220	237	159-289	0.184	10014809 EPA200.8	102010 ICP-MS	Acceptable
Antimony	WP	1005	242	237	159-289	0.82949	10014003 EPA200.7	102015 ICP-AES	Acceptable
Arsenic	WP	1010	432	434	363-509	0.164	10014003 EPA200.7	102015 ICP-AES	Acceptable
Arsenic	WP	1010	496	434	363-509	2.45901	10014809 EPA200.8	102010 ICP-MS	Check for Error
Barium	WP	1015	1560	1530	1300-1730	0.69637	10014003 EPA200.7	102015 ICP-AES	Acceptable
Beryllium	WP	1020	356	402	341-454	2.23	10014003 EPA200.7	102015 ICP-AES	Check for Error
Boron	WP	1025	414	452	390-546	2.08	1014809 EPA200.8	102010 ICP-MS	Check for Error
Cadmium	WP	1030	561	540	461-613	0.94488	10014003 EPA200.7	102015 ICP-AES	Acceptable
Chromium	WP	1040	586	579	504-655	0.23904	10014003 EPA200.7	102015 ICP-AES	Acceptable
Cobalt	WP	1050	581	564	496-632	0.74561	10014003 EPA200.7	102015 ICP-AES	Acceptable
Copper	WP	1055	786	780	709-855	0.16460	10014003 EPA200.7	102015 ICP-AES	Acceptable
Iron	WP	1070	160	160	137-187	0.242	10014003 EPA200.7	102015 ICP-AES	Acceptable
Lead	WP	1075	2740	2650	2330-2950	0.98039	10014003 EPA200.7	102015 ICP-AES	Acceptable

Performance Summary

APG Customer	3655	Labratory Address	Nelac Point Of Contact	WP October 2003 WP #3010	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003

Product: Trace Metals **WP Lot Number: 37208-37209** **APG+ Lot Number: 36544-36545**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Lead	WP	1075	2820	2650	2330-2950	1.76470	10014809 EPA200.8	102010 ICP-MS	Acceptable
Manganese	WP	1090	1580	1570	1410-1740	0.18083	10014003 EPA200.7	102015 ICP-AES	Acceptable
Mercury	WP	1095	25.7	25.5	19.2-31.7	0.14423	10036609 EPA245.1	101010 CVAAS	Acceptable
Mercury	WP	1095	23.8	25.5	19.2-31.7	0.769	10014809 EPA200.8	102010 ICP-MS	Acceptable
Molybdenum	WP	1100	209	207	176-238	0.19607	10014003 EPA200.7	102015 ICP-AES	Acceptable
Nickel	WP	1105	410	418	374-469	0.75	10014003 EPA200.7	102015 ICP-AES	Acceptable
Selenium	WP	1140	1976	1920	1530-2220	0.92173	10014809 EPA200.8	102010 ICP-MS	Acceptable
Selenium	WP	1140	1910	1920	1530-2220	0.34782	10014003 EPA200.7	102015 ICP-AES	Acceptable
Silver	WP	1150	185	176	151-202	1.05386	10014003 EPA200.7	102015 ICP-AES	Acceptable
Strontium	WP	1160	297	298	254-342	0.068	10014809 EPA200.8	102015 ICP-MS	Acceptable
Thallium	WP	1165	593	558	449-648	1.32930	10014809 EPA200.8	102010 ICP-MS	Acceptable
Thallium	WP	1165	574	558	449-648	0.75528	10014003 EPA200.7	102015 ICP-AES	Acceptable
Tin	WP	1175	1370	1540	1220-1870	1.57	1014809 EPA200.8	102010 ICP-MS	Acceptable
Titanium	WP	1180	183	198	170-223	1.57	10014809 EPA200.8	102015 ICP-MS	Acceptable

Performance Summary

APG Customer	3655	Labratory Address	Nelac Point Of Contact	WP October 2003 WP #3010	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003
APG NIST/NVLAP 200384-0		Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003

Product: Trace Metals **WP Lot Number: 37208-37209** **APG+ Lot Number: 36544-36545**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Vanadium	WP	1185	2500	2500	2260-2750	0	10014003 EPA200.7	102015 ICP-AES	Acceptable
Zinc	WP	1190	148	140	120-162	1.00574	10014003 EPA200.7	102015 ICP-AES	Acceptable

Performance Summary

APG Customer	3655	Labratory Address	Nelac Point Of Contact	WP October 2003 WP #3010	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003

Product: Volatiles **WP Lot Number: 37304** **APG+ Lot Number: 37305**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
1,1,1-Trichloroethane	WP	5160	51.1	50.6	32.7-66.8	0.24647	10107207 EPA624	151010 GC-MS	Acceptable
1,1,2,2-Tetrachloroethane	WP	5110	154	153	90.3-213	0.09756	10107207 EPA624	151010 GC-MS	Acceptable
1,1,2-Trichloroethane	WP	5165	133	148	99.9-193	0.839	10107207 EPA624	151010 GC-MS	Acceptable
1,1-Dichloroethane	WP	4630	<0.5			0.839	10107207 EPA624	151010 GC-MS	Acceptable
1,1-Dichloroethene	WP	4640	58.8	52.8	28.5-81.3	0.44318	10107207 EPA624	151010 GC-MS	Acceptable
1,2-Dichloroethane	WP	4635	25.8	27.4	19.1-36.9	0.741	10107207 EPA624	151010 GC-MS	Acceptable
1,2-Dichloropropane	WP	4655	110	107	72-136	0.56074	10107207 EPA624	151010 GC-MS	Acceptable
Bromodichloromethane	WP	4395	30.5	29.1	20.5-38	0.44520	10107207 EPA624	151010 GC-MS	Acceptable
Bromoform	WP	4400	61.3	64.5	42.3-88.2	0.523	10107207 EPA624	151010 GC-MS	Acceptable
Bromomethane	WP	4950	<0.5			0.523	10107207 EPA624	151010 GC-MS	Acceptable
Carbon tetrachloride	WP	4455	20.3	18	11.4-25.5	0.80851	10107207 EPA624	151010 GC-MS	Acceptable
Chlorobenzene	WP	4475	14.9	14.4	10.7-18.1	0.40322	10107207 EPA624	151010 GC-MS	Acceptable
Chloroethane	WP	4485	<0.5			0.40322	10107207 EPA624	151010 GC-MS	Acceptable
Chloroform	WP	4505	49.3	48.4	33.6-62.1	0.29535	10107207 EPA624	151010 GC-MS	Acceptable

Performance Summary

APG Customer 3655	Labratory Address	Nelac Point Of Contact		WP October 2003 WP #3010
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/26/2003
Primary AA E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2003
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/14/2003

Product: Volatiles **WP Lot Number: 37304** **APG+ Lot Number: 37305**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Chloromethane	WP	4960	<0.5			0.29535	10107207 EPA624	151010 GC-MS	Acceptable
Dibromochloromethane	WP	4575	38.8	37.7	24.9-50	0.31026	10107207 EPA624	151010 GC-MS	Acceptable
Methylene chloride	WP	4975	52.2	65.1	41.1-89.4	1.63	10107207 EPA624	151010 GC-MS	Acceptable
Tetrachloroethene	WP	5115	56.8	52.8	34.9-67.6	1.00917	10107207 EPA624	151010 GC-MS	Acceptable
trans-1,2-Dichloroethene	WP	4700	182	184	119-250	0.0913	10107207 EPA624	151010 GC-MS	Acceptable
trans-1,3-Dichloropropene	WP	4685	<0.5			0.0913	10107207 EPA624	151010 GC-MS	Acceptable
Trichloroethene	WP	5170	56.7	55	35.7-71.1	0.55932	10107207 EPA624	151010 GC-MS	Acceptable
Trichlorofluoromethane	WP	5175	<0.5			0.55932	10107207 EPA624	151010 GC-MS	Acceptable
Vinyl Chloride	WP	5235	<0.5			0.55932	10107207 EPA624	151010 GC-MS	Acceptable

Analytical Products Group, Inc.

PERFORMANCE REPORT

WP Performance Summary

April 2004 WP

APG Customer Code: 3655

Analytical Management Lab
Suite B
15130 South Keeler
Olathe, KS 66062

Laboratory of
EXCELLENCE

NVLAP®
LAB CODE 200384-0

Study Certification
April 2004 WP

I have reviewed the statistical analysis 154 which was performed on April 2004 WP. The statistical analysis yielded results which are consistent with NIST and NELAC Requirements.

Thomas V. Coyner
Technical Director

I have reviewed these results and find them to meet the appropriate NELAC and NIST Requirements.

Quality Assurance Manager

This report was produced at
Analytical Products Group, Inc
2730 Washington Blvd.
Belpre, Ohio 45714

This report has been produced in accordance with Analytical Products Group, Inc. ISO 9001:2000 Certification (CERT-9513-2003-AQ-HOU-RAB) issued by Det Norske Vertitas.

Water Pollution (WP) Proficiency Testing Program Report

Introduction:

The Analytical Products Group, Inc. Proficiency Testing Program has been designed to evaluate your laboratory's performance against other participating laboratories on the same set of standards. This report has been specifically prepared for your organization to assist you in ensuring that your procedures are in-control. Therefore, in most cases sample levels are not designed to challenge the detection limits of the method.

The Analytical Products Group Proficiency Testing Program is certified to meet all of the following requirements:

- For applicable analytes, this Proficiency Testing Program is accredited by NIST according to the National Voluntary Laboratory Accreditation Program (Handbook 150-19) for Chemical Calibrations: Providers of Proficiency Testing (NVLAP Code 200384-0)

- Analytical Products Group, Inc. Quality System is registered with DNVCertification to meet the ISO 9001 standard to design, manufacture, and distribute chemical reference materials and operate Proficiency Testing Programs (Certificate # 98-HOU-AQ-9513)

- For applicable analytes, this Proficiency Testing Program is certified to meet all requirements as specified in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).

Please note that this report contains data that are not covered by the NVLAP accredition. This report must not be used to claim product certification, approval, or endorsement by NVLAP, NIST, or any agency of the federal government.

Report Layout

This report contains the following sub-reports:

Introduction -This section is the general introduction to the Analytical Products Group, Inc. Proficiency Testing Program as well as all of the certifications that the organization maintains.

Terms & Definitions - Defines all of the terms used throughout the report. This is an excellent section to refer to first if you have any questions.

Laboratory Benchmark Report - This report presents an overall summary of the study as well as your performance relative to all other study participants.

Comment Report - Contains comments submitted to Analytical Products Group by the program participants or comments by Analytical Products Group to program participants concerning unusual characteristics of the data set or problems with standards which could influence the interpretation of the data. Please review this section thoroughly before examining your data.

Exception Report - This report is a summary of all of the analytes that resulted in an Evaluation of "Not Acceptable" for your laboratory. The Exception Report is designed to very quickly identify for your laboratory the areas that may require your immediate attention.

Performance Evaluation Summary Report - Provides a summary of all of your data for each analyte. This summary is formatted similar to the US EPA DMR-QA Reports.

Detail Report- This report lists all the details of your data. Also included in this section are useful graphs indicating the APG Assigned Value, the Mean, the Acceptance Limits and your Reported Value. Another graph included in this section shows your Z score for the analyte of interest.

The goal of this report is to provide your laboratory with the information you need to monitor and improve your quality. If you have any questions or feedback, please give us a call (800-272-4442). Thank you for choosing Analytical Products Group as your proficiency testing provider.

Sincerely,

Gregg Frazier
General Manager

Terms & Definitions

Average Acceptable Data - This value is the average percent of data that is within the EPA Acceptance Limits for this study. The Average Acceptable Data is reported for all laboratories as a whole as well as your laboratory as an individual. These values can be found in the Laboratory Benchmark Report.

Average Percentile for All Parameters - This is your laboratory's rank on each parameter and the related rank for that parameter. The average percentile is the average of this data across all parameters reported. It is a different measure of performance than percent acceptable data since it is affected by your relative performance on each parameter. The report provides an average for this measure for all laboratories reporting as well as your laboratory's performance.

Analyte - Name of the analyte of interest as found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) or NELAC Field of Testing List.

NELAC Analyte Code - This value is the code given to the analyte by NELAC. These codes can be found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) or the NELAC Fields of Testing List.

APG Assigned Value - This value is the calculated True Value of the standard based upon the actual composition of the standard in most cases.

Z-Score - This figure corresponds to how far your Reported Value was from the EPA Mean, reported in terms of EPA Standard Deviation units. As a point of reference, the US EPA Acceptance Limits are plus or minus three (3) EPA Standard Deviations from the EPA Mean.

APG Certified Value - This value is the result of the quality control testing performed on the sample by Analytical Products Group before the sample is shipped to the customer. Through this testing, the samples are traced against a NIST Standard Reference Material where available or an external standard if a NIST Standard Reference Material is not available. The reported uncertainty of this value is expressed as two times the standard deviation (s) divided by the square root of the number of samples (n) tested (i.e. 2s/vn). This quality control testing meets the requirements as specified by the NIST National Voluntary Laboratory Accreditation Program (NVLAP) Handbook 150-19 (Chemical Calibration: Providers of Proficiency Testing).

Acceptance Range - For applicable analytes, this range is calculated based upon the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045). For the Water Pollution Program (WP), EPA Acceptance Limits are defined as ± three (3) EPA Standard Deviations from the EPA Mean.

Mean - If the analyte is included in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then this value is the estimated mean value calculated per the Criteria Document. If the analyte is not included in the Criteria Document, then this value is the Study Mean.

Study Standard Deviation - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Standard Deviation is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Your Ranking - This is your ranking based on your Z Score for the analyte of interest among all possible rankings. It is important to note that several laboratories can achieve the same rank.

Terms & Definitions (continued)

Evaluation - This value is determined by the Acceptance Limits and the Reported Value. Evaluation may have one of following values:

Value	Description
Acceptable	The Reported Value was within the Acceptable Limits or a less than (<) value was reported AND zero (0) was included in the Acceptance Limit Range AND the numeric part of the Reported Value was less than the upper limit of the Acceptance Limit.
Not Acceptable	The Reported Value is outside of the Acceptance Limits or a less than (<) value was reported AND zero (0) is not included in the Acceptance Limits range or the numeric part of the Reported Value was greater than the upper limit of the Acceptance Limit or a greater than value (>) was reported.
Check for Error	The Reported Value is between two (2) and three (3) Standard Deviations from the Mean.
Not Evaluated	The Reported Value was a text value which could not be evaluated.
Blank	No data was reported for this analyte [the analyte was not spiked].

Inter-Laboratory Statistics - This data is a summary of all of the data from all study participants for the analyte of interest. Inter-Laboratory Statistics are only reported if a minimum number of laboratories analyzed and reported data for the analyte of interest.

Product Level - Often referred to as 'Program'. This value indicates which program the analyte of interest is a part. For Water Pollution Studies (WP), the only values that Program will have are 'WP' or 'APG+'. If the analyte and range are consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then the Program value will have a value of 'WP' and if this is not the case then the Program value will be 'APG+', indicating that the analyte and/or range was specified by Analytical Products Group.

Product Type - To the right of each analyte in the performance summary is a code that designates the scope of the APG's accreditation for that analyte. An analyte designated as "NE" is applicable to NELAC and EPA requirements and ranges. An analyte designated as "N" is applicable to NELAC requirements and ranges. An analyte designated as "E" is applicable to EPA requirements and ranges. An analyte designated as "X" or without a designation is not covered by APG's accreditations.

Rank on Percent Acceptable Data - This is your laboratories rank out of all possible ranks in the current study based on percent acceptable data. It is important to note that several laboratories can achieve the same rank.

Rank on Average Percentile Data - This value is your laboratories rank out of all possible ranks based on Your Ranking converted to a percentile. It is important to note that several laboratories can achieve the same rank.

Laboratory Pass Percentage - This is the percentage of study participants that reported values within the Acceptance Limits for the analyte of interest.

Solids Standards - Solids standards are individually prepared, therefore, each standard has a unique APG Assigned Value.

Terms & Definitions (continued)

Reported Value - This value is the result that was reported by your organization for the particular analyte. Every effort has been made to ensure correct data entry. Should you find a data entry error, please notify Analytical Products Group, Inc. and we will correct the error and re-issue the report. If the Reported Value is reported as a less than (<) figure, then the only way that the Evaluation will result in an "Acceptable" value is if the numeric part of the Reported Value (i.e. 8 if the Report Value were <8) is less than the upper limit of the Acceptance Range AND zero (0) is included in the Acceptance Range. Any other non-numeric value (i.e. BDL, ND, >10, etc.) cannot be evaluated and will return an Evaluation result of "No Evaluation". This method of evaluation is consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).

Number of Acceptable Data Points - This value is the number of data points reported by your laboratory that were within the Acceptance Limits.

Weighted Rank on Percent Acceptable Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percent Acceptable Data is the weighted rank of your laboratory based on Percent Acceptable Data and the number of analytes reported.

Weight Rank on Percentile Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percentile Data is the weighted rank of your laboratory based on Rank on Average Percentile Data and the number of analytes reported.

Study Mean - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Mean is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Laboratory Benchmark Report

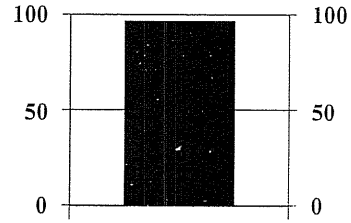
Customer Code: 3655 **April 2004 WP**

Summary of Ranking Data

Summary of National Ranking Data

Total Number of Laboratories Reporting:	231
Average Percent Acceptable Data:	96.53%
Average Percentile for all Parameters:	53.99%

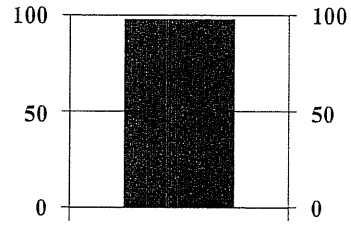
National Avg % Acceptable
96.5 %



Summary of Laboratory Ranking:

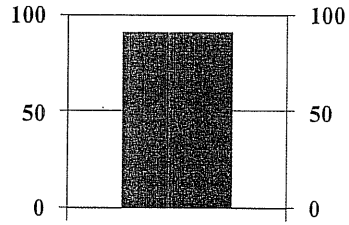
Total Data Points Reported:	225
Number of Acceptable Data Points:	220
Percent Acceptable Data:	97.78%
Average Percentile for all Parameters:	54.58%

Laboratory Avg % Acceptable
97.78%



Rank on Percent Acceptable Data:	20 of 84
Rank on Average Percentile Data:	103 of 196
Weighted Rank on Percent Acceptable Data:	19 of 119
Weighted Rank on Average Percentile Data:	18 of 221

Weighted Rank Avg Percentile
91.9 %



Comment Report

There are no comments to report.

Exception Report

Product: Hexavalent Chromium — **Exception Reporting** — **Units: ug/L**

Analyte	Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Chromium, Hexavalent	WP	1045	74.0	55.7	45.3-64.1	6.19	10162206 EPA7196		Not Acceptable

Product: Trace Metals — **Exception Reporting** — **Units: ug/L**

Analyte		Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Cobalt	NE	WP	1050	479	410	360-460	4.13	10014809 EPA200.8		Not Acceptable
Copper	NE	WP	1055	123	108	94.9-122	3.38	10014809 EPA200.8		Not Acceptable
Manganese	NE	WP	1090	1034	928	834-1030	3.10	10014809 EPA200.8		Not Acceptable
Molybdenum	NE	WP	1100	595	472	405-540	5.45	10014809 EPA200.8		Not Acceptable

Performance Summary

Performance Summary

APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP April 2004 WP #4004
Study Print Date 06/01/2004
Study Shipment Date 04/01/2004
Study Closing Date 05/14/2004

Product: Acid Extractables **WP Lot Number: 39187** **APG+ Lot Number: 39192** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
2,4,5-Trichlorophenol	N	WP	6835	133	169	60.0-215 86.4-190	0.194	10107401 EPA625		Acceptable
2,4,6-Trichlorophenol	N	WP	6840	83.0	107	35.2-142 53.0-124	0.305	10107401 EPA625		Acceptable
2,4-Dichlorophenol	N	WP	6000	<10				10107401 EPA625		Acceptable
2,4-Dimethylphenol	N	WP	6130	<10				10107401 EPA625		Acceptable
2,4-Dinitrophenol	N	WP	6175	87.9	89.1	0.000-123 17.6-102	1.32	10107401 EPA625		Acceptable
2-Chlorophenol	N	WP	5800	77.7	99.6	29.2-125 45.3-109	0.0250	10107401 EPA625		Acceptable
2-Methyl-4,6-Dinitrophenol	N	WP	6360	92.8	107	16.6-150 38.8-128	0.432	10107401 EPA625		Acceptable
2-Nitrophenol	N	WP	6490	76.8	95.0	28.6-125 44.6-109	0.0125	10107401 EPA625		Acceptable
4-Chloro-3-methylphenol	N	WP	5700	40.1	45.9	16.6-59.1 23.7-52.1	0.311	10107401 EPA625		Acceptable
4-Nitrophenol	N	WP	6500	30.0	40.1	0.000-61.4 -2.20-48.6	0.535	10107401 EPA625		Acceptable
Pentachlorophenol	N	WP	6605	140	168	46.7-232 78.2-202	0.000	10107401 EPA625		Acceptable
Phenol	N	WP	6625	107	167	0.000-223 7.00-180	0.309	10107401 EPA625		Acceptable

Performance Summary

APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP April 2004 WP #4004
Study Print Date 06/01/2004
Study Shipment Date 04/01/2004
Study Closing Date 05/14/2004

Product: Aluminum High Level **WP Lot Number: 39231** **APG+ Lot Number: 39232** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aluminum High Level	X	WP	1000	0.351	0.350	0.298-0.404	0.000	10014003		Acceptable
						0.316-0.386		EPA200.7		

Performance Summary

APG Customer 3655	Laboratory Address	Nelac Point Of Contact	**WP April 2004 WP #4004**
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date 06/01/2004**
Primary AA E-10254	15130 South Keeler	QA Director	**Study Shipment Date 04/01/2004**
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date 05/14/2004**

Product: Base Neutral Extractables **WP Lot Number: 39388** **APG+ Lot Number: 39446** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
1,2,4-Trichlorobenzene	N	WP	5155	<10				10107401 EPA625		Acceptable
1,2-Dichlorobenzene	N	WP	4610	37.6	54.2	24.4-52.6 29.1-47.9	0.191	10107401 EPA625		Acceptable
1,3-Dichlorobenzene	N	WP	4615	48.6	74.6	31.0-66.4 36.9-60.5	0.0169	10107401 EPA625		Acceptable
1,4-Dichlorobenzene	N	WP	4620	37.0	53.2	18.9-55.1 24.9-49.1	0.000	10107401 EPA625		Acceptable
2,4-Dinitrotoluene (2,4-DNT)	N	WP	6185	142	170	73.2-227 98.8-201	0.312	10107401 EPA625		Acceptable
2,6-Dinitrotoluene (2,6-DNT)	N	WP	6190	95.0	115	44.0-151 61.9-134	0.151	10107401 EPA625		Acceptable
2-Chloronaphthalene	N	WP	5795	39.8	52.9	20.7-63.4 27.8-56.2	0.309	10107401 EPA625		Acceptable
3,3'-Dichlorobenzidine	N	WP	5945	<10				10107401 EPA625		Acceptable
4-Bromophenyl phenyl ether	N	WP	5660	<10				10107401 EPA625		Acceptable
4-Chlorophenyl phenyl ether	N	WP	5825	<10				10107401 EPA625		Acceptable
Acenaphthene	N	WP	5500	37.5	47.2	18.5-60.3 25.5-53.3	0.273	10107401 EPA625		Acceptable
Acenaphthylene	N	WP	5505	49.4	58.7	22.9-72.3 31.1-64.1	0.218	10107401 EPA625		Acceptable
Anthracene	N	WP	5555	75.4	102	36.3-132 52.2-116	0.541	10107401 EPA625		Acceptable

Performance Summary

APG Customer 3655	Laboratory Address	Nelac Point Of Contact	WP April 2004 WP #4004
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date 06/01/2004
Primary AA E-10254	15130 South Keeler	QA Director	Study Shipment Date 04/01/2004
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date 05/14/2004

Product: Base Neutral Extractables **WP Lot Number: 39388** **APG+ Lot Number: 39446** **Units: ug/L**

Analyte	Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Benzidine	N WP	5595	<10				10107401 EPA625		Acceptable
Benzo(a)anthracene	N WP	5575	41.5	49.3	27.0-58.8 32.3-53.5	0.264	10107401 EPA625		Acceptable
Benzo(a)pyrene	N WP	5580	24.9	33.3	5.06-44.3 11.6-37.8	0.0305	10107401 EPA625		Acceptable
Benzo(b)fluoranthene	N WP	5585	<10				10107401 EPA625		Acceptable
Benzo(g,h,i)perylene	N WP	5590	<10				10107401 EPA625		Acceptable
Benzo(k)fluoranthene	N WP	5600	<10				10107401 EPA625		Acceptable
Bis(2-Chloroethoxy)methane	N WP	5760	<10				10107401 EPA625		Acceptable
Bis(2-Chloroethyl)ether	N WP	5765	<10				10107401 EPA625		Acceptable
Bis(2-Chloroisopropyl)ether	N WP	5780	<10				10107401 EPA625		Acceptable
Bis(2-Ethylhexyl)phthalate	N WP	6255	47.9	62.1	17.1-90.1 29.2-78.0	0.467	10107401 EPA625		Acceptable
Butyl benzyl phthalate	N WP	5670	52.5	63.2	4.62-90.8 18.9-76.5	0.333	10107401 EPA625		Acceptable
Chrysene	N WP	5855	35.7	41.2	18.1-53.2 24.0-47.4	0.000	10107401 EPA625		Acceptable
Dibenzo(a,h)anthracene	N WP	5895	<10				10107401 EPA625		Acceptable

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		**WP April 2004 WP #4004**
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**06/01/2004**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**04/01/2004**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**05/14/2004**

Product: Base Neutral Extractables **WP Lot Number: 39388** **APG+ Lot Number: 39446** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Diethyl phthalate	N	WP	6070	122	175	0.000-268 29.6-220	0.0629	10107401 EPA625		Acceptable
Dimethyl phthalate	N	WP	6135	72.9	90.2	0.000-138 11.5-113	0.427	10107401 EPA625		Acceptable
Di-n-butyl phthalate	N	WP	5925	99.0	153	16.5-214 49.4-181	0.488	10107401 EPA625		Acceptable
Di-n-octyl phthalate	N	WP	6200	38.2	43.4	9.96-65.1 19.1-55.9	0.0762	10107401 EPA625		Acceptable
Fluoranthene	N	WP	6265	115	174	72.7-225 98.2-200	1.34	10107401 EPA625		Acceptable
Fluorene	N	WP	6270	83.4	114	47.7-146 64.1-130	0.823	10107401 EPA625		Acceptable
Hexachlorobenzene	N	WP	6275	43.4	50.8	22.2-65.8 29.4-58.6	0.0824	10107401 EPA625		Acceptable
Hexachlorobutadiene	N	WP	4835	51.8	74.5	11.9-90.4 25.0-77.4	0.0458	10107401 EPA625		Acceptable
Hexachlorocyclopentadiene	N	WP	6285	95.8	118	0.000-151 13.9-124	0.989	10107401 EPA625		Acceptable
Hexachloroethane	N	WP	4840	39.4	58.9	6.95-73.4 18.0-62.4	0.0721	10107401 EPA625		Acceptable
Indeno(1,2,3-cd)pyrene	N	WP	6315	<10				10107401 EPA625		Acceptable
Isophorone	N	WP	6320	64.0	73.6	26.7-97.3 38.4-85.6	0.169	10107401 EPA625		Acceptable
Naphthalene	N	WP	5005	87.6	144	34.7-187 60.0-162	0.918	10107401 EPA625		Acceptable

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact	**WP April 2004 WP #4004**	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**06/01/2004**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**04/01/2004**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**05/14/2004**

Product: Base Neutral Extractables **WP Lot Number: 39388** **APG+ Lot Number: 39446** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Nitrobenzene (NB)	N	WP	5015	58.0	96.3	31.6-126 47.2-110	1.31	10107401 EPA625		Acceptable
N-nitrosodimethylamine	N	WP	6530	<10				10107401 EPA625		Acceptable
N-nitrosodi-n-propylamine	N	WP	6545	<10				10107401 EPA625		Acceptable
N-nitrosodiphenylamine	N	WP	6535	<10				10107401 EPA625		Acceptable
Phenanthrene	N	WP	6615	102	159	52.3-210 78.4-184	1.10	10107401 EPA625		Acceptable
Pyrene	N	WP	6665	90.6	131	43.6-177 65.6-154	0.874	10107401 EPA625		Acceptable

Performance Summary

APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP April 2004 WP #4004
Study Print Date 06/01/2004
Study Shipment Date 04/01/2004
Study Closing Date 05/14/2004

Product: BTEX **WP Lot Number: 39150** **APG+ Lot Number: 39155** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Benzene	NE	WP	4375	48.0	47.9	34.5-61.7 39.0-57.2	0.0221	10107207 EPA624		Acceptable
Ethylbenzene	NE	WP	4765	46.8	48.9	32.6-63.9 37.9-58.7	0.287	10107207 EPA624		Acceptable
m-Xylene / p-Xylene	X	WP	5240	203	216	146-289 170-266	0.630	10107207 EPA624		Acceptable
o-Xylene	X	WP	5250	38.2	37.4	26.5-47.3 29.9-43.9	0.374	10107207 EPA624		Acceptable
Toluene	NE	WP	5140	13.4	12.6	8.72-16.3 9.98-15.0	0.714	10107207 EPA624		Acceptable
Total Xylenes	N	WP	5260	241	253	146-337 177-305	0.000	10107207 EPA624		Acceptable

Product: Demand **WP Lot Number: 39280** **APG+ Lot Number: 39281** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Biochemical Oxygen Demand	NE	WP	1530	26.5	30.9	15.3-46.5 20.5-41.3	0.848	10075602 EPA405.1		Acceptable
Carbonaceous BOD	NE	WP	1555	23.5	26.7	11.9-41.5 16.9-36.5	0.651	20027401 SM18thED5210B		Acceptable
Chemical Oxygen Demand	NE	WP	1565	48.7	49.8	33.5-62.3 38.3-57.5	0.167	10076003 EPA410.1		Acceptable
Total Organic Carbon	NE	WP	2040	19.2	19.7	16.3-23.0 17.5-21.9	0.450	10078407 EPA415.1		Acceptable

Performance Summary

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APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

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913-829-0101 x26

WP April 2004 WP #4004
Study Print Date 06/01/2004
Study Shipment Date 04/01/2004
Study Closing Date 05/14/2004

Product: Fluoride **WP Lot Number: 39254** **APG+ Lot Number: 39255** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Fluoride	NE	WP	1730	0.721	0.718	0.600-0.832 0.639-0.793	0.130	10053006 EPA300.0		Acceptable

Product: Herbicides **WP Lot Number: 39188** **APG+ Lot Number: 39193** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
2,4,5-T	N	WP	8655	2.17	8.58	0.952-12.5 2.87-10.6	2.36	10105609 EPA615		**Check for Error**
2,4,5-TP (silvex)	N	WP	8650	1.02	2.33	0.0219-3.56 0.612-2.97	1.31	10105609 EPA615		Acceptable
2,4-D	N	WP	8545	1.34	5.59	0.000-8.77 1.18-7.26	1.89	10105609 EPA615		Acceptable
Dicamba	N	WP	8595	1.45	8.36	0.751-12.1 2.64-10.2	2.63	10105609 EPA615		**Check for Error**
Dinoseb	X	WP	8620	3.21	4.42	0.000-6.02 0.0800-4.84	0.630	10105609 EPA615		Acceptable

Product: Hexavalent Chromium **WP Lot Number: 39229** **APG+ Lot Number: 39230** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Chromium, Hexavalent		WP	1045	74.0	55.7	45.3-64.1 48.5-60.9	6.19	10162206 EPA7196		**Not Acceptable**

Performance Summary

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APG Customer 3655	Laboratory Address	Nelac Point Of Contact	WP April 2004 WP #4004
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date 06/01/2004
Primary AA E-10254	15130 South Keeler	QA Director	Study Shipment Date 04/01/2004
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date 05/14/2004

Product: Minerals **WP Lot Number: 39219-39220** **APG+ Lot Number: 39221-39222** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Alkalinity as CaCO3	NE	WP	1505	42.4	38.9	33.9-44.6 35.6-42.8	1.80	10054805 EPA310.1		Acceptable
Calcium	NE	WP	1035	8.21	7.79	6.62-9.18 7.05-8.75	0.726	10014003 EPA200.7		Acceptable
Chloride	NE	WP	1575	88.6	90.5	82.0-98.9 84.8-96.0	0.641	10057406 EPA325.3		Acceptable
Chloride	NE	WP	1575	86.8	90.5	82.0-98.9 84.8-96.0	1.28	10053006 EPA300.0		Acceptable
Conductivity	NE	WP	1610	432	434	400-468 411-457	0.175	10006209 EPA120.1		Acceptable
Magnesium	NE	WP	1085	12.4	12.2	10.6-13.7 11.2-13.2	0.387	10014003 EPA200.7		Acceptable
Potassium	NE	WP	1125	33.8	34.1	29.5-38.8 31.1-37.3	0.256	10014003 EPA200.7		Acceptable
Sodium	NE	WP	1155	31.6	31.4	28.1-35.0 29.3-33.9	0.000	10014003 EPA200.7		Acceptable
Sulfate	NE	WP	2000	13.5	14.3	10.7-17.5 11.8-16.4	0.526	10053006 EPA300.0		Acceptable
Sulfate	NE	WP	2000	11.6	14.3	10.7-17.5 11.8-16.4	2.19	10073606 EPA375.4		**Check for Error**
Total Dissolved Solids	NE	WP	1705	174	229	164-282 184-262	2.49	10009208 EPA160.1		**Check for Error**
Total Hardness as CaCO3	NE	WP	1755	71.8	69.7	61.9-78.1 64.6-75.4	0.664	20003401 SM1819ED2340B		Acceptable

Performance Summary

APG Customer 3655	Laboratory Address	Nelac Point Of Contact	WP April 2004 WP #4004
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date 06/01/2004
Primary AA E-10254	15130 South Keeler	QA Director	Study Shipment Date 04/01/2004
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date 05/14/2004

Product: Nutrients **WP Lot Number: 39265-39266** **APG+ Lot Number: 39268-39269** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Ammonia Nitrogen as N	NE	WP	1515	0.966	1.11	0.777-1.46 0.894-1.35	1.36	10064207 EPA350.3		Acceptable
Nitrate Nitrogen as N	NE	WP	1810	4.36	4.28	3.36-5.12 3.65-4.83	0.410	10067808 EPA353.3		Acceptable
Nitrate Nitrogen as N	NE	WP	1810	3.78	4.28	3.36-5.12 3.65-4.83	1.57	10053006 EPA300.0		Acceptable
Orthophosphate as P	NE	WP	1870	4.40	4.57	3.90-5.28 4.13-5.05	0.830	10070607 EPA365.3		Acceptable
Orthophosphate as P	NE	WP	1870	4.27	4.57	3.90-5.28 4.13-5.05	1.40	10053006 EPA300.0		Acceptable
Total Kjeldahl Nitrogen	NE	WP	1795		19.2	14.0-23.8 15.6-22.2				Not Reported
Total Phosphorus as P	NE	WP	1910	4.81	6.00	4.56-7.02 4.97-6.61	2.39	10070801 EPA365.3		**Check for Error**

Product: Oil & Grease **WP Lot Number: 39198** **APG+ Lot Number: 39199** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Oil & Grease	NE	WP	1860	31.0	42.5	28.2-48.3 31.6-45.0	2.19	10127409 EPA1664		**Check for Error**

Performance Summary

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APG Customer	3655	Laboratory Address	Nelac Point Of Contact	**WP April 2004 WP #4004**	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**06/01/2004**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**04/01/2004**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**05/14/2004**

Product: PCB's in Oil **WP Lot Number: 39196-39197** **Units: mg/kg**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aroclor 1016/1242 in Oil Sample 1	NE	WP	8880	<1				10103603 EPA608		Acceptable
Aroclor 1016/1242 in Oil Sample 2	NE	WP	8880	34.0	43.0	8.05-57.3 16.3-49.1	0.158	10103603 EPA608		Acceptable
Aroclor 1254 in Oil Sample 1	NE	WP	8905	25.8	26.8	2.84-40.8 9.14-34.5	0.632	10103603 EPA608		Acceptable
Aroclor 1254 in Oil Sample 2	NE	WP	8905	<1				10103603 EPA608		Acceptable
Aroclor 1260 in Oil Sample 1	NE	WP	8910	<1				10103603 EPA608		Acceptable
Aroclor 1260 in Oil Sample 2	NE	WP	8910	<1				10103603 EPA608		Acceptable

Performance Summary

APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP April 2004 WP #4004
Study Print Date 06/01/2004
Study Shipment Date 04/01/2004
Study Closing Date 05/14/2004

Product: PCB's in Water **WP Lot Number: 39275-39276** **APG+ Lot Number: 39277** **Units: ug/L**

Analyte	Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aroclor 1016/1242 Sample 1	NE WP	8880	<0.2				10103603 EPA608		Acceptable
Aroclor 1016/1242 Sample 2	NE WP	8880	<0.2				10103603 EPA608		Acceptable
Aroclor 1232 Sample 1	NE WP	8890	<0.2				10103603 EPA608		Acceptable
Aroclor 1232 Sample 2	NE WP	8890	<0.2				10103603 EPA608		Acceptable
Aroclor 1248 Sample 1	NE WP	8900	3.83	4.59	1.91-6.47 2.67-5.71	0.473	10103603 EPA608		Acceptable
Aroclor 1248 Sample 2	NE WP	8900	<0.2				10103603 EPA608		Acceptable
Aroclor 1254 Sample 1	NE WP	8905	<0.2				10103603 EPA608		Acceptable
Aroclor 1254 Sample 2	NE WP	8905	<0.2				10103603 EPA608		Acceptable
Aroclor 1260 Sample 1	NE WP	8910	<0.2				10103603 EPA608		Acceptable
Aroclor 1260 Sample 2	NE WP	8910	2.07	2.49	1.17-3.31 1.53-2.95	0.478	10103603 EPA608		Acceptable

Performance Summary

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APG Customer	3655	Laboratory Address	Nelac Point Of Contact	**WP April 2004 WP #4004**	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**06/01/2004**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**04/01/2004**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**05/14/2004**

Product: Pesticides (NELAC) **WP Lot Number: 39241** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
alpha-BHC	N	WP	7110	4.90	5.34	2.03-7.36 2.92-6.48	0.225	10103603 EPA608		Acceptable
beta-BHC	N	WP	7115	3.00	3.01	0.983-4.51 1.57-3.93	0.425	10103603 EPA608		Acceptable
delta-BHC	N	WP	7105	19.3	20.4	4.92-28.7 8.88-24.7	0.631	10103603 EPA608		Acceptable
Endosulfan I	N	WP	7510	7.56	8.40	3.46-12.1 4.91-10.7	0.160	10103603 EPA608		Acceptable
Endosulfan II	N	WP	7515	37.7	35.6	6.59-57.5 15.1-49.1	0.660	10103603 EPA608		Acceptable
Endosulfan sulfate	N	WP	7520	7.63	8.49	2.10-13.5 3.99-11.6	0.0842	10103603 EPA608		Acceptable
Endrin	N	WP	7540	5.78	5.97	2.37-8.38 3.37-7.37	0.410	10103603 EPA608		Acceptable
Endrin aldehyde	N	WP	7530	12.4	13.1	4.28-19.2 6.74-16.7	0.282	10103603 EPA608		Acceptable
gamma-BHC	N	WP	7120	16.7	17.2	5.43-25.0 8.68-21.7	0.460	10103603 EPA608		Acceptable
Methoxychlor	N	WP	7810	17.4	17.7	7.46-24.9 10.4-22.0	0.414	10103603 EPA608		Acceptable

Performance Summary

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APG Customer 3655	Laboratory Address	Nelac Point Of Contact	**WP April 2004 WP #4004**
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date 06/01/2004**
Primary AA E-10254	15130 South Keeler	QA Director	**Study Shipment Date 04/01/2004**
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date 05/14/2004**

Product: Pesticides (WP) **WP Lot Number: 39240** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
4,4'-DDD	NE	WP	7355	2.17	2.20	1.07-3.08 1.41-2.75	0.269	10103603 EPA608		Acceptable
4,4'-DDE	NE	WP	7360	3.63	3.67	1.74-5.01 2.29-4.47	0.459	10103603 EPA608		Acceptable
4,4'-DDT	NE	WP	7365	2.73	2.83	1.29-3.84 1.71-3.41	0.399	10103603 EPA608		Acceptable
Aldrin	NE	WP	7025	0.170	0.236	0.0567-0.322 0.101-0.277	0.430	10103603 EPA608		Acceptable
Dieldrin	NE	WP	7470	0.127	0.150	0.0742-0.212 0.0972-0.189	0.699	10103603 EPA608		Acceptable
Heptachlor	NE	WP	7685	0.282	0.333	0.0953-0.459 0.156-0.398	0.0824	10103603 EPA608		Acceptable
Heptachlor epoxide (beta)	NE	WP	7690	0.490	0.518	0.262-0.670 0.330-0.602	0.353	10103603 EPA608		Acceptable

Product: pH **WP Lot Number: 39409** **APG+ Lot Number: 39410** **Units: Units**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
pH	NE	WP	1900	9.10	9.01	8.74-9.28 8.83-9.19	1.00	10008409 EPA150.1		Acceptable

Performance Summary

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APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP April 2004 WP #4004
Study Print Date 06/01/2004
Study Shipment Date 04/01/2004
Study Closing Date 05/14/2004

Product: Polynuclear Aromatic Hydrocarbons-PAH **WP Lot Number: 39394** **APG+ Lot Number: 39395** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Acenaphthene	X	WP	5500	12.4	15.5	5.18-18.0 7.32-15.9	0.374	10107401 EPA625		Acceptable
Acenaphthylene	X	WP	5505	8.70	10.1	3.70-11.5 4.98-10.2	0.862	10107401 EPA625		Acceptable
Anthracene	X	WP	5555	1.20	1.42	0.530-1.63 0.714-1.45	0.656	10107401 EPA625		Acceptable
Benzo(a)anthracene	X	WP	5575	<0.2				10107401 EPA625		Acceptable
Benzo(a)pyrene	X	WP	5580	<0.2				10107401 EPA625		Acceptable
Benzo(b)fluoranthene	X	WP	5585	<0.2				10107401 EPA625		Acceptable
Benzo(g,h,i)perylene	X	WP	5590	<0.2				10107401 EPA625		Acceptable
Benzo(k)fluoranthene	X	WP	5600	<0.2				10107401 EPA625		Acceptable
Chrysene	X	WP	5855	<0.2				10107401 EPA625		Acceptable
Dibenzo(a,h)anthracene	X	WP	5895	<0.2				10107401 EPA625		Acceptable
Fluoranthene	X	WP	6265	1.02	0.937	0.423-1.09 0.535-0.983	2.33	10107401 EPA625		**Check for Error**
Fluorene	X	WP	6270	12.6	14.4	5.62-16.2 7.38-14.4	0.966	10107401 EPA625		Acceptable

Performance Summary

APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP April 2004 WP #4004
Study Print Date 06/01/2004
Study Shipment Date 04/01/2004
Study Closing Date 05/14/2004

Product: Polynuclear Aromatic Hydrocarbons-PAH **WP Lot Number: 39394** **APG+ Lot Number: 39395** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Indeno(1,2,3-cd)pyrene	X	WP	6315	<0.2				10107401 EPA625		Acceptable
Naphthalene	X	WP	5005	5.62	7.64	2.27-8.83 3.37-7.73	0.0642	10107401 EPA625		Acceptable
Phenanthrene	X	WP	6615	1.01	0.954	0.334-1.25 0.486-1.09	1.45	10107401 EPA625		Acceptable
Pyrene	X	WP	6665	0.96	0.916	0.383-1.08 0.500-0.968	1.93	10107401 EPA625		Acceptable

Product: Solids **WP Lot Number: 90726** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Dissolved Solids		WP	1705	423	417	310-548 350-508	0.151	10009208 EPA160.1		Acceptable
Total Solids		WP	1950	490	513	307-747 380-674	0.504	10009800 EPA160.3		Acceptable
Total Suspended Solids	NE	WP	1960	98.0	96.2	74.8-104 79.7-99.1	1.77	10009606 EPA160.2		Acceptable

Product: Total Chlordane **WP Lot Number: 39169** **APG+ Lot Number: 39170** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Chlordane (Total)	NE	WP	7250	2.45	2.68	1.18-3.81 1.61-3.37	0.0913	10103603 EPA608		Acceptable

Performance Summary

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APG Customer 3655	Laboratory Address	Nelac Point Of Contact	WP April 2004 WP #4004
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date 06/01/2004
Primary AA E-10254	15130 South Keeler	QA Director	Study Shipment Date 04/01/2004
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date 05/14/2004

Product: Total Cyanide WP Lot Number: 39186 APG+ Lot Number: 39191 Units: mg/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Cyanide	NE	WP	1645	0.937	0.936	0.652-1.20 0.744-1.11	0.109	10060409 EPA335.2		Acceptable

Product: Total Phenolics WP Lot Number: 39185 APG+ Lot Number: 39190 Units: mg/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Phenolics	NE	WP	1905	0.214	0.360	0.124-0.353 0.162-0.314	0.630	10079808 EPA420.2		Acceptable

Product: Total Residual Chlorine WP Lot Number: 39223 APG+ Lot Number: 39224 Units: mg/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Residual Chlorine	NE	WP	1940	4.6	4.37	3.41-4.85 3.65-4.61	1.97	10059208 EPA330.4		Acceptable

Product: Toxaphene WP Lot Number: 39171 APG+ Lot Number: 39172 Units: ug/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Toxaphene	N	WP	8250	3.05	4.65	0.509-8.95 1.91-7.55	1.19	10103603 EPA608		Acceptable

Product: TPH Diesel (DRO) WP Lot Number: 39225 APG+ Lot Number: 39226 Units: mg/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
TPH Diesel (DRO)	X	WP	9369	0.69	2.89	0.278-3.44 0.808-2.91	2.22	10173203 EPA8015		Check for Error

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact	**WP April 2004 WP #4004**	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**06/01/2004**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**04/01/2004**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**05/14/2004**

Product: TPH Gasoline (GRO) **WP Lot Number: 39227** **APG+ Lot Number: 39228** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
TPH Gasoline (GRO)	X	WP	9408	2.63	3.68	0.000-2440 -1340-1680	0.224	10173203 EPA8015		Acceptable

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		**WP April 2004 WP #4004**
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**06/01/2004**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**04/01/2004**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**05/14/2004**

Product: Trace Metals **WP Lot Number: 39149-39380** **APG+ Lot Number: 39154-39381** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aluminum	NE	WP	1000	2610	2570	2210-2910 2330-2790	0.431	10014003 EPA200.7		Acceptable
Aluminum	NE	WP	1000	2430	2570	2210-2910 2330-2790	1.12	10014809 EPA200.8		Acceptable
Antimony	NE	WP	1005	540	494	345-595 387-553	1.68	10014003 EPA200.7		Acceptable
Antimony	NE	WP	1005	571	494	345-595 387-553	2.42	10014809 EPA200.8		**Check for Error**
Arsenic	NE	WP	1010	501	495	415-580 442-552	0.145	10014003 EPA200.7		Acceptable
Arsenic	NE	WP	1010	485	495	415-580 442-552	0.435	10014809 EPA200.8		Acceptable
Barium	N	WP	1015	439	438	379-501 399-481	0.0490	10014003 EPA200.7		Acceptable
Barium	N	WP	1015	450	438	379-501 399-481	0.490	10014809 EPA200.8		Acceptable
Beryllium	NE	WP	1020	323	344	292-389 308-372	1.06	10014003 EPA200.7		Acceptable
Beryllium	NE	WP	1020	351	344	292-389 308-372	0.683	10014809 EPA200.8		Acceptable
Boron	X	WP	1025	621	659	571-753 601-723	1.35	10156000 EPA6020		Acceptable
Boron	X	WP	1025	621	659	571-753 601-723	1.35	10014809 EPA200.8		Acceptable
Cadmium	NE	WP	1030	175	169	144-193 152-184	0.852	10014003 EPA200.7		Acceptable

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact	**WP April 2004 WP #4004**	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**06/01/2004**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**04/01/2004**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**05/14/2004**

Product: Trace Metals **WP Lot Number: 39149-39380** **APG+ Lot Number: 39154-39381** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Cadmium	NE	WP	1030	171	169	144-193 152-184	0.365	10014809 EPA200.8		Acceptable
Chromium	NE	WP	1040	76.9	74.0	62.1-85.6 66.1-81.7	0.767	10014003 EPA200.7		Acceptable
Chromium	NE	WP	1040	78.0	74.0	62.1-85.6 66.1-81.7	1.05	10014809 EPA200.8		Acceptable
Cobalt	NE	WP	1050	435	410	360-460 377-443	1.50	10014003 EPA200.7		Acceptable
Cobalt	NE	WP	1050	479	410	360-460 377-443	4.13	10014809 EPA200.8		**Not Acceptable**
Copper	NE	WP	1055	111	108	94.9-122 99.1-117	0.676	10014003 EPA200.7		Acceptable
Copper	NE	WP	1055	123	108	94.9-122 99.1-117	3.38	10014809 EPA200.8		**Not Acceptable**
Iron	NE	WP	1070	504	500	439-569 461-547	0.000	10014003 EPA200.7		Acceptable
Iron	NE	WP	1070	533	500	439-569 461-547	1.35	10014809 EPA200.8		Acceptable
Lead	NE	WP	1075	452	439	381-495 400-476	0.733	10014003 EPA200.7		Acceptable
Lead	NE	WP	1075	475	439	381-495 400-476	1.94	10014809 EPA200.8		Acceptable
Manganese	NE	WP	1090	970	928	834-1030 866-998	1.16	10014003 EPA200.7		Acceptable
Manganese	NE	WP	1090	1034	928	834-1030 866-998	3.10	10014809 EPA200.8		**Not Acceptable**

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		**WP April 2004 WP #4004**
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**06/01/2004**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**04/01/2004**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**05/14/2004**

Product: Trace Metals **WP Lot Number: 39149-39380** **APG+ Lot Number: 39154-39381** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Mercury	NE	WP	1095	5.17	5.42	3.98-6.84 4.46-6.36	0.503	10036609 EPA245.1		Acceptable
Mercury	NE	WP	1095	4.73	5.42	3.98-6.84 4.46-6.36	1.43	10014809 EPA200.8		Acceptable
Molybdenum	NE	WP	1100	487	472	405-540 428-518	0.625	10014003 EPA200.7		Acceptable
Molybdenum	NE	WP	1100	595	472	405-540 428-518	5.45	10014809 EPA200.8		**Not Acceptable**
Nickel	NE	WP	1105	455	435	389-488 406-472	0.970	10014003 EPA200.7		Acceptable
Nickel	NE	WP	1105	488	435	389-488 406-472	2.97	10014809 EPA200.8		**Check for Error**
Selenium	NE	WP	1140	317	320	251-372 272-352	0.250	10014003 EPA200.7		Acceptable
Selenium	NE	WP	1140	320	320	251-372 272-352	0.400	10014809 EPA200.8		Acceptable
Silver	NE	WP	1150	125	119	102-137 107-131	1.03	10014003 EPA200.7		Acceptable
Silver	NE	WP	1150	118	119	102-137 107-131	0.172	10014809 EPA200.8		Acceptable
Strontium	NE	WP	1160	220	224	191-257 202-246	0.360	10014809 EPA200.8		Acceptable
Strontium	NE	WP	1160	220	224	191-257 202-246	0.360	10014809 EPA200.8		Acceptable
Thallium	NE	WP	1165	193	187	149-219 160-208	0.763	10014003 EPA200.7		Acceptable

Performance Summary

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APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP April 2004 WP #4004
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	06/01/2004
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	04/01/2004
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	05/14/2004

Product: Trace Metals **WP Lot Number: 39149-39380** **APG+ Lot Number: 39154-39381** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Thallium	NE	WP	1165	201	187	149-219 160-208	1.44	10014809 EPA200.8		Acceptable
Tin	N	WP	1175	3940	3570	2810-4350 3070-4090	1.41	10014809 EPA200.8		Acceptable
Tin	N	WP	1175	3940	3570	2810-4350 3070-4090	1.41	10014809 EPA200.8		Acceptable
Titanium	NE	WP	1180	93.6	105	89.3-119 94.0-114	2.08	10014809 EPA200.8		**Check for Error**
Titanium	NE	WP	1180	93.6	105	89.3-119 94.0-114	2.08	10014809 EPA200.8		**Check for Error**
Vanadium	NE	WP	1185	809	800	720-878 746-852	0.380	10014003 EPA200.7		Acceptable
Vanadium	NE	WP	1185	769	800	720-878 746-852	1.14	10014809 EPA200.8		Acceptable
Zinc	NE	WP	1190	490	452	398-511 416-492	1.91	10014003 EPA200.7		Acceptable
Zinc	NE	WP	1190	490	452	398-511 416-492	1.91	10014809 EPA200.8		Acceptable

Performance Summary

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APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP April 2004 WP #4004
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	06/01/2004
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	04/01/2004
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	05/14/2004

Product: Volatiles **WP Lot Number: 39249** **APG+ Lot Number: 39250** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
1,1,1-Trichloroethane	NE	WP	5160	42.6	46.4	30.0-61.2 35.2-56.0	0.577	10107207 EPA624		Acceptable
1,1,2,2-Tetrachloroethane	N	WP	5110	121	132	77.8-184 95.6-166	0.565	10107207 EPA624		Acceptable
1,1,2-Trichloroethane	N	WP	5165	123	133	89.9-173 104-160	0.647	10107207 EPA624		Acceptable
1,1-Dichloroethane	X	WP	4630	<2				10107207 EPA624		Acceptable
1,1-Dichloroethene	N	WP	4640	59.3	59.2	32.2-90.8 42.0-81.0	0.225	10107207 EPA624		Acceptable
1,2-Dichloroethane	NE	WP	4635	28.7	36.1	25.0-48.3 28.9-44.5	2.06	10107207 EPA624		**Check for Error**
1,2-Dichloropropane	N	WP	4655	107	116	77.9-148 89.8-136	0.517	10107207 EPA624		Acceptable
Bromodichloromethane	NE	WP	4395	28.5	34.0	24.0-44.3 27.4-41.0	1.68	10107207 EPA624		Acceptable
Bromoform	NE	WP	4400	40.0	49.9	32.5-68.3 38.4-62.4	1.74	10107207 EPA624		Acceptable
Bromomethane	N	WP	4950	<2				10107207 EPA624		Acceptable
Carbon tetrachloride	NE	WP	4455	24.8	28.0	17.5-39.6 21.1-35.9	1.01	10107207 EPA624		Acceptable
Chlorobenzene	NE	WP	4475	18.7	19.2	14.0-24.3 15.8-22.6	0.292	10107207 EPA624		Acceptable
Chloroethane	N	WP	4485	<2				10107207 EPA624		Acceptable

Performance Summary

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APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP April 2004 WP #4004
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	06/01/2004
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	04/01/2004
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	05/14/2004

Product: Volatiles **WP Lot Number: 39249** **APG+ Lot Number: 39250** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Chloroform	NE	WP	4505	36.1	40.7	28.3-52.3 32.3-48.3	1.05	10107207 EPA624		Acceptable
Chloromethane	N	WP	4960	<2				10107207 EPA624		Acceptable
Dibromochloromethane	NE	WP	4575	27.4	31.7	20.9-42.1 24.5-38.5	1.16	10107207 EPA624		Acceptable
Methylene chloride	NE	WP	4975	49.3	61.3	38.7-84.2 46.3-76.7	1.61	10107207 EPA624		Acceptable
Tetrachloroethene	NE	WP	5115	64.5	67.0	44.4-85.6 51.2-78.8	0.0727	10107207 EPA624		Acceptable
trans-1,2-Dichloroethene	N	WP	4700	146	155	99.5-211 118-192	0.484	10107207 EPA624		Acceptable
trans-1,3-Dichloropropene	N	WP	4685	<2				10107207 EPA624		Acceptable
Trichloroethene	NE	WP	5170	45.3	50.4	32.7-65.1 38.1-59.7	0.665	10107207 EPA624		Acceptable
Trichlorofluoromethane	N	WP	5175	<2				10107207 EPA624		Acceptable
Vinyl Chloride	N	WP	5235	<2				10107207 EPA624		Acceptable

Analytical Products Group, Inc.

PERFORMANCE REPORT

WP Performance Summary

October 2004 WP

APG Customer Code: 3655

Analytical Management Lab
Suite B
15130 South Keeler
Olathe, KS 66062

Laboratory of EXCELLENCE

NVLAP®
LAB CODE 200384-0

Study Certification
October 2004 WP

I have reviewed the statistical analysis 220 which was performed on October 2004 WP. The statistical analysis yielded results which are consistent with NIST and NELAC Requirements.

Thomas V. Coyner
Technical Director

I have reviewed these results and find them to meet the appropriate NELAC and NIST Requirements.

Quality Assurance Manager

This report was produced at
Analytical Products Group, Inc
2730 Washington Blvd.
Belpre, Ohio 45714

This report has been produced in accordance with Analytical Products Group, Inc. ISO 9001:2000 Certification (CERT-9513-2003-AQ-HOU-RAB) issued by Det Norske Vertitas.

Water Pollution (WP) Proficiency Testing Program Report

Introduction:

The Analytical Products Group, Inc. Proficiency Testing Program has been designed to evaluate your laboratory's performance against other participating laboratories on the same set of standards. This report has been specifically prepared for your organization to assist you in ensuring that your procedures are in-control. Therefore, in most cases sample levels are not designed to challenge the detection limits of the method.

The Analytical Products Group Proficiency Testing Program is certified to meet all of the following requirements:

- For applicable analytes, this Proficiency Testing Program is accredited by NIST according to the National Voluntary Laboratory Accreditation Program (Handbook 150-19) for Chemical Calibrations: Providers of Proficiency Testing (NVLAP Code 200384-0)

- Analytical Products Group, Inc. Quality System is registered with DNVCertification to meet the ISO 9001 standard to design, manufacture, and distribute chemical reference materials and operate Proficiency Testing Programs (Certificate # 98-HOU-AQ-9513)

- For applicable analytes, this Proficiency Testing Program is certified to meet all requirements as specified in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).

Please note that this report contains data that are not covered by the NVLAP accredition. This report must not be used to claim product certification, approval, or endorsement by NVLAP, NIST, or any agency of the federal government.

Report Layout

This report contains the following sub-reports:

Introduction -This section is the general introduction to the Analytical Products Group, Inc. Proficiency Testing Program as well as all of the certifications that the organization maintains.

Terms & Definitions - Defines all of the terms used throughout the report. This is an excellent section to refer to first if you have any questions.

Laboratory Benchmark Report - This report presents an overall summary of the study as well as your performance relative to all other study participants.

Comment Report - Contains comments submitted to Analytical Products Group by the program participants or comments by Analytical Products Group to program participants concerning unusual characteristics of the data set or problems with standards which could influence the interpretation of the data. Please review this section thoroughly before examining your data.

Exception Report - This report is a summary of all of the analytes that resulted in an Evaluation of "Not Acceptable" for your laboratory. The Exception Report is designed to very quickly identify for your laboratory the areas that may require your immediate attention.

Performance Evaluation Summary Report - Provides a summary of all of your data for each analyte. This summary is formatted similar to the US EPA DMR-QA Reports.

Detail Report- This report lists all the details of your data. Also included in this section are useful graphs indicating the APG Assigned Value, the Mean, the Acceptance Limits and your Reported Value. Another graph included in this section shows your Z score for the analyte of interest.

The goal of this report is to provide your laboratory with the information you need to monitor and improve your quality. If you have any questions or feedback, please give us a call (800-272-4442). Thank you for choosing Analytical Products Group as your proficiency testing provider.

Sincerely,

Gregg Frazier
General Manager

Terms & Definitions

Average Acceptable Data - This value is the average percent of data that is within the EPA Acceptance Limits for this study. The Average Acceptable Data is reported for all laboratories as a whole as well as your laboratory as an individual. These values can be found in the Laboratory Benchmark Report.

Average Percentile for All Parameters - This is your laboratory's rank on each parameter and the related rank for that parameter. The average percentile is the average of this data across all parameters reported. It is a different measure of performance than percent acceptable data since it is affected by your relative performance on each parameter. The report provides an average for this measure for all laboratories reporting as well as your laboratory's performance.

Analyte - Name of the analyte of interest as found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) or NELAC Field of Testing List.

NELAC Analyte Code - This value is the code given to the analyte by NELAC. These codes can be found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) or the NELAC Fields of Testing List.

APG Assigned Value - This value is the calculated True Value of the standard based upon the actual composition of the standard in most cases.

Z-Score - This figure corresponds to how far your Reported Value was from the EPA Mean, reported in terms of EPA Standard Deviation units. As a point of reference, the US EPA Acceptance Limits are plus or minus three (3) EPA Standard Deviations from the EPA Mean.

APG Certified Value - This value is the result of the quality control testing performed on the sample by Analytical Products Group before the sample is shipped to the customer. Through this testing, the samples are traced against a NIST Standard Reference Material where available or an external standard if a NIST Standard Reference Material is not available. The reported uncertainty of this value is expressed as two times the standard deviation (s) divided by the square root of the number of samples (n) tested (i.e. 2s/vn). This quality control testing meets the requirements as specified by the NIST National Voluntary Laboratory Accreditation Program (NVLAP) Handbook 150-19 (Chemical Calibration: Providers of Proficiency Testing).

Acceptance Range - For applicable analytes, this range is calculated based upon the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045). For the Water Pollution Program (WP), EPA Acceptance Limits are defined as ± three (3) EPA Standard Deviations from the EPA Mean.

Mean - If the analyte is included in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then this value is the estimated mean value calculated per the Criteria Document. If the analyte is not included in the Criteria Document, then this value is the Study Mean.

Study Standard Deviation - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Standard Deviation is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Your Ranking - This is your ranking based on your Z Score for the analyte of interest among all possible rankings. It is important to note that several laboratories can achieve the same rank.

Terms & Definitions (continued)

Evaluation - This value is determined by the Acceptance Limits and the Reported Value. Evaluation may have one of following values:

Value	Description
Acceptable	The Reported Value was within the Acceptable Limits or a less than (<) value was reported AND zero (0) was included in the Acceptance Limit Range AND the numeric part of the Reported Value was less than the upper limit of the Acceptance Limit.
Not Acceptable	The Reported Value is outside of the Acceptance Limits or a less than (<) value was reported AND zero (0) is not included in the Acceptance Limits range or the numeric part of the Reported Value was greater than the upper limit of the Acceptance Limit or a greater than value (>) was reported.
Check for Error	The Reported Value is between two (2) and three (3) Standard Deviations from the Mean.
Not Evaluated	The Reported Value was a text value which could not be evaluated.
Blank	No data was reported for this analyte [the analyte was not spiked].

Inter-Laboratory Statistics - This data is a summary of all of the data from all study participants for the analyte of interest. Inter-Laboratory Statistics are only reported if a minimum number of laboratories analyzed and reported data for the analyte of interest.

Product Level - Often referred to as 'Program'. This value indicates which program the analyte of interest is a part. For Water Pollution Studies (WP), the only values that Program will have are 'WP' or 'APG+'. If the analyte and range are consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then the Program value will have a value of 'WP' and if this is not the case then the Program value will be 'APG+', indicating that the analyte and/or range was specified by Analytical Products Group.

Product Type - To the right of each analyte in the performance summary is a code that designates the scope of the APG's accreditation for that analyte. An analyte designated as "NE" is applicable to NELAC and EPA requirements and ranges. An analyte designated as "N" is applicable to NELAC requirements and ranges. An analyte designated as "E" is applicable to EPA requirements and ranges. An analyte designated as "X" or without a designation is not covered by APG's accreditations.

Rank on Percent Acceptable Data - This is your laboratories rank out of all possible ranks in the current study based on percent acceptable data. It is important to note that several laboratories can achieve the same rank.

Rank on Average Percentile Data - This value is your laboratories rank out of all possible ranks based on Your Ranking converted to a percentile. It is important to note that several laboratories can achieve the same rank.

Laboratory Pass Percentage - This is the percentage of study participants that reported values within the Acceptance Limits for the analyte of interest.

Solids Standards - Solids standards are individually prepared, therefore, each standard has a unique APG Assigned Value.

Terms & Definitions (continued)

Reported Value - This value is the result that was reported by your organization for the particular analyte. Every effort has been made to ensure correct data entry. Should you find a data entry error, please notify Analytical Products Group, Inc. and we will correct the error and re-issue the report. If the Reported Value is reported as a less than (<) figure, then the only way that the Evaluation will result in an "Acceptable" value is if the numeric part of the Reported Value (i.e. 8 if the Report Value were <8) is less than the upper limit of the Acceptance Range AND zero (0) is included in the Acceptance Range. Any other non-numeric value (i.e. BDL, ND, >10, etc.) cannot be evaluated and will return an Evaluation result of "No Evaluation". This method of evaluation is consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).

Number of Acceptable Data Points - This value is the number of data points reported by your laboratory that were within the Acceptance Limits.

Weighted Rank on Percent Acceptable Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percent Acceptable Data is the weighted rank of your laboratory based on Percent - Acceptable Data and the number of analytes reported.

Weight Rank on Percentile Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percentile Data is the weighted rank of your laboratory based on Rank on Average Percentile Data and the number of analytes reported.

Study Mean - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Mean is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Laboratory Benchmark Report

Customer Code: 3655

October 2004 WP

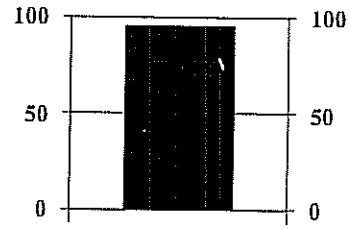
Summary of Ranking Data

Summary of National Ranking Data

Total Number of Laboratories Reporting:	294
Average Percent Acceptable Data:	95.92%
Average Percentile for all Parameters:	54.04%

National Avg % Acceptable

95.9 %

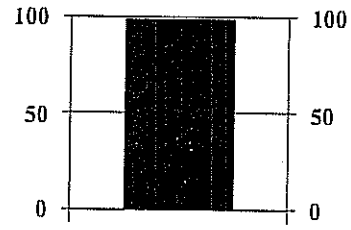


Summary of Laboratory Ranking:

Total Data Points Reported:	238
Number of Acceptable Data Points:	235
Percent Acceptable Data:	98.74%
Average Percentile for all Parameters:	55.74%
Rank on Percent Acceptable Data:	13 of 84
Rank on Average Percentile Data:	124 of 248
Weighted Rank on Percent Acceptable Data:	12 of 127
Weighted Rank on Average Percentile Data:	12 of 264

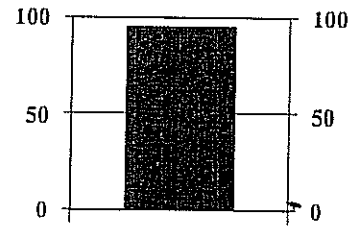
Laboratory Avg % Acceptable

98.74%



Weighted Rank Avg Percentile

95.5 %



Comment Report

There are no comments to report.

Exception Report

Analytical Products Group, Inc.
2730 Washington Boulevard • Belpre, Ohio 45714 • 800.272.4442 • Fax 740.423.5588 • www.APGQA.com

Product: Demand — Exception Reporting — Units: mg/L

Analyte		Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code / Method Desc.	Technology Code / Technology Desc.	Evaluation
Total Organic Carbon	NE	WP	2040	34.0	43.4	36.2-50.1	3.92	10078203 / EPA415.1	402010 / IR	Not Acceptable

Product: Minerals — Exception Reporting — Units: mg/L

Analyte		Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code / Method Desc.	Technology Code / Technology Desc.	Evaluation
Potassium	NE	WP	1125	18.0	21.4	18.4-24.5	3.33	10013408 / EPA200.7	102015 / ICP-AES	Not Acceptable

Product: Polynuclear Aromatic Hydrocarbons-PAH — Exception Reporting — Units: ug/L

Analyte		Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code / Method Desc.	Technology Code / Technology Desc.	Evaluation
Pyrene	X	WP	6665	0.50	1.04	0.539-1.14	3.40	10107401 / EPA625	151010 / GC-MS	Not Acceptable

Performance Summary

Performance Summary

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APG Customer 3655	Laboratory Address	Nelac Point Of Contact	WP October 2004 WP #4010
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date 11/23/2004
Primary AA E-10254	15130 South Keeler	QA Director	Study Shipment Date 10/01/2004
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date 11/15/2004

Product: Acid Extractables **WP Lot Number: 40773** **APG+ Lot Number: 40774** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
2,4,5-Trichlorophenol	N	WP	6835	<10				10107401 EPA625	151010 GC-MS	Acceptable
2,4,6-Trichlorophenol	N	WP	6840	28.7	31.9	12.0-41.2 16.9-36.3	0.431	10107401 EPA625	151010 GC-MS	Acceptable
2,4-Dichlorophenol	N	WP	6000	59.3	71.5	25.8-87.5 36.1-77.3	0.252	10107401 EPA625	151010 GC-MS	Acceptable
2,4-Dimethylphenol	N	WP	6130	122	143	31.6-187 57.2-161	0.502	10107401 EPA625	151010 GC-MS	Acceptable
2,4-Dinitrophenol	N	WP	6175	34.1	46.3	0.000-74.8 11.9-62.3	0.238	10107401 EPA625	151010 GC-MS	Acceptable
2-Chlorophenol	N	WP	5800	65.2	82.9	24.7-105 38.1-91.3	0.0376	10107401 EPA625	151010 GC-MS	Acceptable
2-Methyl-4,6-Dinitrophenol	N	WP	6360	89.2	116	16.7-164 41.2-140	0.0488	10107401 EPA625	151010 GC-MS	Acceptable
2-Nitrophenol	N	WP	6490	39.8	46.5	14.6-60.4 22.2-52.8	0.301	10107401 EPA625	151010 GC-MS	Acceptable
4-Chloro-3-methylphenol	N	WP	5700	135	161	63.5-207 87.2-183	0.000	10107401 EPA625	151010 GC-MS	Acceptable
4-Nitrophenol	N	WP	6500	44.2	71.4	0.000-101 0.000-80.9	0.201	10107401 EPA625	151010 GC-MS	Acceptable
Pentachlorophenol	N	WP	6605	93.1	106	27.2-147 47.1-127	0.312	10107401 EPA625	151010 GC-MS	Acceptable
Phenol	N	WP	6625	<10				10107401 EPA625	151010 GC-MS	Acceptable

Performance Summary

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APG Customer	3655	Laboratory Address	Nelac Point Of Contact	WP October 2004 WP #4010	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/23/2004
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2004
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/15/2004

Product: Aluminum High Level **WP Lot Number: 40668** **APG+ Lot Number: 40669** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aluminum High Level	X	WP	1000	0.657	0.703	0.606-0.781	1.23	10013408	102015	Acceptable
						0.635-0.751		EPA200.7	ICP-AES	

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP October 2004 WP #4010
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/23/2004
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2004
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/15/2004

Product: Base Neutral Extractables **WP Lot Number: 40811** **APG+ Lot Number: 40812** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
1,2,4-Trichlorobenzene	N	WP	5155	73.4	90.3	26.3-108	0.456	10107401	151010	Acceptable
						40.0-94.4		EPA625	GC-MS	
1,2-Dichlorobenzene	N	WP	4610	52.0	62.6	9.01-75.5	0.874	10107401	151010	Acceptable
						20.1-64.5		EPA625	GC-MS	
1,3-Dichlorobenzene	N	WP	4615	67.5	86.0	7.12-102	0.799	10107401	151010	Acceptable
						23.0-86.6		EPA625	GC-MS	
1,4-Dichlorobenzene	N	WP	4620	44.7	56.0	5.76-67.5	0.786	10107401	151010	Acceptable
						16.0-57.2		EPA625	GC-MS	
2,4-Dinitrotoluene (2,4-DNT)	N	WP	6185	36.6	39.2	12.6-51.3	0.728	10107401	151010	Acceptable
						19.0-44.8		EPA625	GC-MS	
2,6-Dinitrotoluene (2,6-DNT)	N	WP	6190	124	135	51.6-179	0.427	10107401	151010	Acceptable
						72.8-157		EPA625	GC-MS	
2-Chloronaphthalene	N	WP	5795	69.3	77.3	30.7-89.8	0.924	10107401	151010	Acceptable
						40.5-79.9		EPA625	GC-MS	
3,3'-Dichlorobenzidine	N	WP	5945	<10				10107401	151010	Acceptable
								EPA625	GC-MS	
4-Bromophenyl phenyl ether	N	WP	5660	<10				10107401	151010	Acceptable
								EPA625	GC-MS	
4-Chlorophenyl phenyl ether	N	WP	5825	<10				10107401	151010	Acceptable
								EPA625	GC-MS	
Acenaphthene	N	WP	5500	53.4	58.3	22.5-74.5	0.565	10107401	151010	Acceptable
						31.2-65.8		EPA625	GC-MS	
Acenaphthylene	N	WP	5505	21.2	20.3	7.17-26.6	1.33	10107401	151010	Acceptable
						10.4-23.4		EPA625	GC-MS	
Anthracene	N	WP	5555	34.7	35.6	17.0-46.1	0.639	10107401	151010	Acceptable
						21.9-41.3		EPA625	GC-MS	

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP October 2004 WP #4010
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/23/2004
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2004
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/15/2004

Product: Base Neutral Extractables **WP Lot Number:** 40811 **APG+ Lot Number:** 40812 **Units:** ug/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Benzidine	N	WP	5595	<10				10107401 EPA625	151010 GC-MS	Acceptable
Benzo(a)anthracene	N	WP	5575	34.5	36.2	20.8-42.1 24.4-38.6	0.845	10107401 EPA625	151010 GC-MS	Acceptable
Benzo(a)pyrene	N	WP	5580	30.8	34.6	5.48-46.0 12.2-39.2	0.756	10107401 EPA625	151010 GC-MS	Acceptable
Benzo(b)fluoranthene	N	WP	5585	<10				10107401 EPA625	151010 GC-MS	Acceptable
Benzo(g,h,i)perylene	N	WP	5590	<10				10107401 EPA625	151010 GC-MS	Acceptable
Benzo(k)fluoranthene	N	WP	5600	<10				10107401 EPA625	151010 GC-MS	Acceptable
Bis(2-Chloroethoxy)methane	N	WP	5760	<10				10107401 EPA625	151010 GC-MS	Acceptable
Bis(2-Chloroethyl)ether	N	WP	5765	<10				10107401 EPA625	151010 GC-MS	Acceptable
Bis(2-Chloroisopropyl)ether	N	WP	5780	<10				10107401 EPA625	151010 GC-MS	Acceptable
Bis(2-Ethylhexyl)phthalate	N	WP	6255	132	189	22.5-278 65.0-235	0.424	10107401 EPA625	151010 GC-MS	Acceptable
Butyl benzyl phthalate	N	WP	5670	72.8	77.6	0.000-124 15.5-102	0.653	10107401 EPA625	151010 GC-MS	Acceptable
Chrysene	N	WP	5855	37.8	37.8	17.0-48.7 22.2-43.4	0.947	10107401 EPA625	151010 GC-MS	Acceptable
Di-n-butyl phthalate	N	WP	5925	115	137	16.1-191 45.6-162	0.377	10107401 EPA625	151010 GC-MS	Acceptable

Performance Summary

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APG Customer 3655	Laboratory Address	Nelac Point Of Contact	WP October 2004 WP #4010
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date 11/23/2004
Primary AA E-10254	15130 South Keeler	QA Director	Study Shipment Date 10/01/2004
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date 11/15/2004

Product: Base Neutral Extractables **WP Lot Number: 40811** **APG+ Lot Number: 40812** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Di-n-octyl phthalate	N	WP	6200	109	152	20.1-220 53.4-187	0.330	10107401 EPA625	151010 GC-MS	Acceptable
Dibenzo(a,h)anthracene	N	WP	5895	<10				10107401 EPA625	151010 GC-MS	Acceptable
Diethyl phthalate	N	WP	6070	31.5	31.8	12.1-47.7 18.0-41.8	0.270	10107401 EPA625	151010 GC-MS	Acceptable
Dimethyl phthalate	N	WP	6135	32.3	34.1	11.5-51.9 18.3-45.1	0.0893	10107401 EPA625	151010 GC-MS	Acceptable
Fluoranthene	N	WP	6265	50.0	52.7	24.4-67.7 31.7-60.5	0.541	10107401 EPA625	151010 GC-MS	Acceptable
Fluorene	N	WP	6270	64.4	70.1	29.5-90.3 39.7-80.1	0.446	10107401 EPA625	151010 GC-MS	Acceptable
Hexachlorobenzene	N	WP	6275	91.7	106	46.4-137 61.4-122	0.00662	10107401 EPA625	151010 GC-MS	Acceptable
Hexachlorobutadiene	N	WP	4835	<10				10107401 EPA625	151010 GC-MS	Acceptable
Hexachlorocyclopentadiene	N	WP	6285	13.0	15.8	0.000-24.3 0.000-19.4	0.697	10107401 EPA625	151010 GC-MS	Acceptable
Hexachloroethane	N	WP	4840	61.0	79.7	8.10-100 23.4-85.0	0.442	10107401 EPA625	151010 GC-MS	Acceptable
Indeno(1,2,3-cd)pyrene	N	WP	6315	<10				10107401 EPA625	151010 GC-MS	Acceptable
Isophorone	N	WP	6320	33.2	36.2	15.1-46.9 20.4-41.6	0.415	10107401 EPA625	151010 GC-MS	Acceptable
N-nitrosodi-n-propylamine	N	WP	6545	<10				10107401 EPA625	151010 GC-MS	Acceptable

Performance Summary

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APG Customer 3655	Laboratory Address	Nelac Point Of Contact	WP October 2004 WP #4010
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date 11/23/2004
Primary AA E-10254	15130 South Keeler	QA Director	Study Shipment Date 10/01/2004
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date 11/15/2004

Product: Base Neutral Extractables **WP Lot Number: 40811** **APG+ Lot Number: 40812** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
N-nitrosodimethylamine	N	WP	6530	<10				10107401 EPA625	151010 GC-MS	Acceptable
N-nitrosodiphenylamine	N	WP	6535	<10				10107401 EPA625	151010 GC-MS	Acceptable
Naphthalene	N	WP	5005	47.4	53.2	15.7-69.8 24.8-60.8	0.510	10107401 EPA625	151010 GC-MS	Acceptable
Nitrobenzene (NB)	N	WP	5015	41.7	49.0	17.2-63.6 24.9-55.9	0.168	10107401 EPA625	151010 GC-MS	Acceptable
Phenanthrene	N	WP	6615	44.9	48.7	24.3-61.3 30.5-55.1	0.341	10107401 EPA625	151010 GC-MS	Acceptable
Pyrene	N	WP	6665	48.5	50.6	16.5-71.8 25.8-62.6	0.467	10107401 EPA625	151010 GC-MS	Acceptable

Performance Summary

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APG Customer	3655	Laboratory Address	Nelac Point Of Contact	**WP October 2004 WP #4010**	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**11/23/2004**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**10/01/2004**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**11/15/2004**

Product: BTEX **WP Lot Number: 40764** **APG+ Lot Number: 40765** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Benzene	NE	WP	4375	9.48	10.0	6.83-13.4	0.569	10107207	151010	Acceptable
						7.92-12.3		EPA624	GC-MS	
Ethylbenzene	NE	WP	4765	22.7	22.2	15.0-28.8	0.348	10107207	151010	Acceptable
						17.3-26.5		EPA624	GC-MS	
m-Xylene / p-Xylene	X	WP	5240	42.9	42.3	27.9-53.1	0.571	10107207	151010	Acceptable
						32.1-48.9		EPA624	GC-MS	
o-Xylene	X	WP	5250	34.3	35.7	22.6-47.0	0.123	10107207	151010	Acceptable
						26.6-43.0		EPA624	GC-MS	
Toluene	NE	WP	5140	17.8	17.3	12.1-22.1	0.422	10107207	151010	Acceptable
						13.8-20.4		EPA624	GC-MS	
Total Xylenes	N	WP	5260	77.2	78.1	44.3-106	0.183	10107207	151010	Acceptable
						54.5-96.1		EPA624	GC-MS	

Product: Demand **WP Lot Number: 40739** **APG+ Lot Number: 40740** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Biochemical Oxygen Demand	NE	WP	1530	65.6	67.9	34.2-102	0.205	10075408	352010	Acceptable
						45.4-90.4		EPA405.1	POT	
Carbonaceous BOD	NE	WP	1555	74.4	58.5	26.2-90.8	1.47	20027401	352010	Acceptable
						37.0-80.0		SM18thED5210B	POT	
Chemical Oxygen Demand	NE	WP	1565	105	110	82.3-128	0.000	10076003	401010	Acceptable
						89.7-120		EPA410.1	COLOR	
Total Organic Carbon	NE	WP	2040	34.0	43.4	36.2-50.1	3.92	10078203	402010	**Not Acceptable**
						38.5-47.7		EPA415.1	IR	

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact	**WP October 2004 WP #4010**	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**11/23/2004**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**10/01/2004**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**11/15/2004**

Product: Fluoride **WP Lot Number: 40675** **APG+ Lot Number: 40676** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Fluoride	NE	WP	1730	2.32	2.51	2.19-2.81	1.75	10053006	251510	Acceptable
						2.29-2.71		EPA300.0	IC	

Product: Herbicides **WP Lot Number: 40775** **APG+ Lot Number: 40776** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
2,4,5-T	N	WP	8655	6.47	6.75	0.645-9.92	0.768	10105609	152010	Acceptable
						2.18-8.38		EPA615	GC-ECD	
2,4,5-TP (silvex)	N	WP	8650	6.19	6.18	0.307-8.36	1.39	10105609	152010	Acceptable
						1.65-7.01		EPA615	GC-ECD	
2,4-D	N	WP	8545	3.98	8.44	0.000-12.7	1.02	10105609	152010	Acceptable
						1.86-10.5		EPA615	GC-ECD	
Dicamba	N	WP	8595	3.31	5.46	0.412-7.99	0.706	10105609	152010	Acceptable
						1.68-6.72		EPA615	GC-ECD	
Dinoseb	X	WP	8620	1.44	1.44	0.000-2.08	1.59	10105609	152010	Acceptable
						0.000-1.63		EPA615	GC-ECD	

Product: Hexavalent Chromium **WP Lot Number: 40696** **APG+ Lot Number: 40697** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Chromium, Hexavalent		WP	1045	406	390	317-449	1.06	10162206	401010	Acceptable
						339-427		EPA7196	COLOR	

Performance Summary

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APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP October 2004 WP #4010
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/23/2004
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2004
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/15/2004

Product: Minerals **WP Lot Number: 40846-40847** **APG+ Lot Number: 40848-40849** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Alkalinity as CaCO3	NE	WP	1505	19.4	20.0	16.3-24.8	0.769	10054601	651010	Acceptable
						17.6-23.4		EPA310.1	TITR	
Calcium	NE	WP	1035	10.4	10.8	9.33-12.6	0.926	10013408	102015	Acceptable
						9.82-12.0		EPA200.7	ICP-AES	
Chloride	NE	WP	1575	88.6	93.9	85.1-103	1.79	10057406	651010	Acceptable
						88.0-99.6		EPA325.3	TITR	
Chloride	NE	WP	1575	89.0	93.9	85.1-103	1.66	10053006	251510	Acceptable
						88.0-99.6		EPA300.0	IC	
Conductivity	NE	WP	1610	383	371	357-417	0.400	10006209	351010	Acceptable
						367-407		EPA120.1	COND	
Magnesium	NE	WP	1085	12.7	14.5	12.6-16.3	2.96	10013408	102015	**Check for Error**
						13.3-15.7		EPA200.7	ICP-AES	
Potassium	NE	WP	1125	18.0	21.4	18.4-24.5	3.33	10013408	102015	**Not Acceptable**
						19.4-23.4		EPA200.7	ICP-AES	
Sodium	NE	WP	1155	19.3	21.6	19.2-24.4	2.91	10013408	102015	**Check for Error**
						20.1-23.5		EPA200.7	ICP-AES	
Sulfate	NE	WP	2000	7.2	8.21	5.59-10.7	1.11	10073606	401010	Acceptable
						6.44-9.84		EPA375.4	COLOR	
Sulfate	NE	WP	2000	7.88	8.21	5.59-10.7	0.306	10053006	251510	Acceptable
						6.44-9.84		EPA300.0	IC	
Total Dissolved Solids	NE	WP	1705	223	239	153-265	0.749	10009004	201010	Acceptable
						172-246		EPA160.1	GRAV	
Total Hardness as CaCO3	NE	WP	1755	78.3	86.9	77.7-96.6	2.83	20003401	102015	**Check for Error**
						80.9-93.5		SM1819ED2340B	ICP-AES	

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact	**WP October 2004 WP #4010**	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**11/23/2004**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**10/01/2004**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**11/15/2004**

Product: Nutrients **WP Lot Number: 40718-40719** **APG+ Lot Number: 40720-40721** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Ammonia Nitrogen as N	NE	WP	1515	5.98	5.50	4.22-6.74 4.64-6.32	1.19	10063806 EPA350.2	401010 COLOR	Acceptable
Ammonia Nitrogen as N	NE	WP	1515	5.97	5.50	4.22-6.74 4.64-6.32	1.16	10064207 EPA350.3	352010 POT	Acceptable
Nitrate Nitrogen as N	NE	WP	1810	6.86	8.00	6.32-9.52 6.85-8.99	1.99	10067808 EPA353.3	401010 COLOR	Acceptable
Nitrate Nitrogen as N	NE	WP	1810	7.28	8.00	6.32-9.52 6.85-8.99	1.20	10053006 EPA300.0	251510 IC	Acceptable
Orthophosphate as P	NE	WP	1870	3.20	3.00	2.56-3.47 2.71-3.31	1.25	10070607 EPA365.3	401010 COLOR	Acceptable
Orthophosphate as P	NE	WP	1870	3.04	3.00	2.56-3.47 2.71-3.31	0.197	10053006 EPA300.0	251510 IC	Acceptable
Total Kjeldahl Nitrogen	NE	WP	1795		19.1	13.9-23.6 15.6-22.0				Not Reported
Total Phosphorus as P	NE	WP	1910	3.59	4.15	3.15-4.87 3.44-4.58	1.47	10070607 EPA365.3	401010 COLOR	Acceptable

Product: Oil & Grease **WP Lot Number: 40793** **APG+ Lot Number: 40794** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Oil & Grease	NE	WP	1860	27.9	25.4	15.5-30.5 18.0-28.0	1.98	10127409 EPA1664	201010 GRAV	Acceptable

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP October 2004 WP #4010
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/23/2004
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2004
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/15/2004

Product: PCB's in Oil — **WP Lot Number: 40791-40792** — **Units: mg/kg**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aroclor 1016/1242 in Oil Sample 1	NE	WP	8880	<1				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1016/1242 in Oil Sample 2	NE	WP	8880	35.5	48.8	9.50-64.2 18.7-55.1	0.154	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1254 in Oil Sample 1	NE	WP	8905	31.2	40.6	6.51-59.0 15.3-50.3	0.183	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1254 in Oil Sample 2	NE	WP	8905	<1				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1260 in Oil Sample 1	NE	WP	8910	<1				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1260 in Oil Sample 2	NE	WP	8910	<1				10103603 EPA608	152010 GC-ECD	Acceptable

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact	**WP October 2004 WP #4010**	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**11/23/2004**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**10/01/2004**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**11/15/2004**

Product: PCB's in Water **WP Lot Number: 40723-40724** **APG+ Lot Number: 40725** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aroclor 1016/1242 Sample 1	NE	WP	8880	<0.5				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1016/1242 Sample 2	NE	WP	8880	5.58	6.31	1.95-8.75 3.09-7.61	0.204	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1232 Sample 1	NE	WP	8890	<0.5				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1232 Sample 2	NE	WP	8890	<0.5				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1248 Sample 1	NE	WP	8900	3.66	4.59	1.91-6.47 2.67-5.71	0.696	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1248 Sample 2	NE	WP	8900	<0.5				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1254 Sample 1	NE	WP	8905	<0.5				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1254 Sample 2	NE	WP	8905	<0.5				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1260 Sample 1	NE	WP	8910	<0.5				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1260 Sample 2	NE	WP	8910	<0.5				10103603 EPA608	152010 GC-ECD	Acceptable

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact	**WP October 2004 WP #4010**	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**11/23/2004**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**10/01/2004**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**11/15/2004**

Product: Pesticides (NELAC) **WP Lot Number: 40883** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
alpha-BHC	N	WP	7110	9.30	11.1	3.90-15.5	0.207	10103603	152010	Acceptable
						5.84-13.6		EPA608	GC-ECD	
beta-BHC	N	WP	7115	3.77	4.28	1.48-6.31	0.149	10103603	152010	Acceptable
						2.28-5.50		EPA608	GC-ECD	
delta-BHC	N	WP	7105	11.0	12.9	3.51-17.4	0.260	10103603	152010	Acceptable
						5.78-15.0		EPA608	GC-ECD	
Endosulfan I	N	WP	7510	3.33	3.81	1.56-5.35	0.190	10103603	152010	Acceptable
						2.19-4.71		EPA608	GC-ECD	
Endosulfan II	N	WP	7515	36.0	38.6	7.04-62.5	0.130	10103603	152010	Acceptable
						16.3-53.3		EPA608	GC-ECD	
Endosulfan sulfate	N	WP	7520	9.58	10.6	2.32-17.0	0.0408	10103603	152010	Acceptable
						4.78-14.6		EPA608	GC-ECD	
Endrin	N	WP	7540	20.7	25.2	8.68-38.2	0.569	10103603	152010	Acceptable
						13.7-33.3		EPA608	GC-ECD	
Endrin aldehyde	N	WP	7530	7.28	6.21	2.04-9.12	1.44	10103603	152010	Acceptable
						3.22-7.94		EPA608	GC-ECD	
gamma-BHC	N	WP	7120	14.8	16.5	5.22-24.0	0.0641	10103603	152010	Acceptable
						8.36-20.8		EPA608	GC-ECD	
Methoxychlor	N	WP	7810	9.28	8.87	3.52-13.0	0.652	10103603	152010	Acceptable
						5.09-11.4		EPA608	GC-ECD	

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP October 2004 WP #4010
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/23/2004
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2004
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/15/2004

Product: Pesticides (WP) **WP Lot Number: 40882** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
4,4'-DDD	NE	WP	7355	2.26	2.72	1.33-3.80	0.754	10103603	152010	Acceptable
						1.75-3.39		EPA608	GC-ECD	
4,4'-DDE	NE	WP	7360	0.541	0.683	0.315-0.930	0.786	10103603	152010	Acceptable
						0.416-0.828		EPA608	GC-ECD	
4,4'-DDT	NE	WP	7365	0.247	0.288	0.113-0.420	0.370	10103603	152010	Acceptable
						0.163-0.369		EPA608	GC-ECD	
Aldrin	NE	WP	7025	0.713	1.27	0.323-1.72	1.32	10103603	152010	Acceptable
						0.556-1.48		EPA608	GC-ECD	
Dieldrin	NE	WP	7470	0.703	0.828	0.425-1.15	0.692	10103603	152010	Acceptable
						0.546-1.03		EPA608	GC-ECD	
Heptachlor	NE	WP	7685	1.37	2.29	0.605-3.19	1.23	10103603	152010	Acceptable
						1.04-2.76		EPA608	GC-ECD	
Heptachlor epoxide (beta)	NE	WP	7690	1.78	2.06	1.05-2.65	0.262	10103603	152010	Acceptable
						1.32-2.38		EPA608	GC-ECD	

Product: pH **WP Lot Number: 40925** **APG+ Lot Number: 40926** **Units: Units**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
pH	NE	WP	1900	5.84	5.81	5.68-5.94	0.688	10008205	352010	Acceptable
						5.72-5.90		EPA150.1	. POT	

Performance Summary

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APG Customer 3655	Laboratory Address	Nelac Point Of Contact	WP October 2004 WP #4010
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date 11/23/2004
Primary AA E-10254	15130 South Keeler	QA Director	Study Shipment Date 10/01/2004
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date 11/15/2004

Product: Polynuclear Aromatic Hydrocarbons-PAH **WP Lot Number:** 40813 **APG+ Lot Number:** 40814 **Units:** ug/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Acenaphthene	X	WP	5500	<0.1				10107401 EPA625	151010 GC-MS	Acceptable
Acenaphthene	X	WP	5500	<0.25				10187607 EPA8310	251020 HPLC-UV	Acceptable
Acenaphthylene	X	WP	5505	<0.1				10107401 EPA625	151010 GC-MS	Acceptable
Acenaphthylene	X	WP	5505	<0.13				10187607 EPA8310	251020 HPLC-UV	Acceptable
Anthracene	X	WP	5555	0.628	1.20	0.343-1.67 0.568-1.45	1.73	10187607 EPA8310	251020 HPLC-UV	Acceptable
Anthracene	X	WP	5555	0.52	1.20	0.343-1.67 0.568-1.45	2.22	10107401 EPA625	151010 GC-MS	**Check for Error**
Benzo(a)anthracene	X	WP	5575	<0.1				10107401 EPA625	151010 GC-MS	Acceptable
Benzo(a)anthracene	X	WP	5575	<0.01				10187607 EPA8310	251020 HPLC-UV	Acceptable
Benzo(a)pyrene	X	WP	5580	<0.1				10107401 EPA625	151010 GC-MS	Acceptable
Benzo(a)pyrene	X	WP	5580	<0.01				10187607 EPA8310	251020 HPLC-UV	Acceptable
Benzo(b)fluoranthene	X	WP	5585	<0.1				10107401 EPA625	151010 GC-MS	Acceptable
Benzo(b)fluoranthene	X	WP	5585	<0.01				10187607 EPA8310	251020 HPLC-UV	Acceptable

Performance Summary

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APG Customer	3655	Laboratory Address	Nelac Point Of Contact	WP October 2004 WP #4010	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/23/2004
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2004
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/15/2004

Product: Polynuclear Aromatic Hydrocarbons-PAH **WP Lot Number:** 40813 **APG+ Lot Number:** 40814 **Units:** ug/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Benzo(g,h,i)perylene	X	WP	5590	<0.1				10107401 EPA625	151010 GC-MS	Acceptable
Benzo(g,h,i)perylene	X	WP	5590	<0.02				10187607 EPA8310	251020 HPLC-UV	Acceptable
Benzo(k)fluoranthene	X	WP	5600	<0.1				10107401 EPA625	151010 GC-MS	Acceptable
Benzo(k)fluoranthene	X	WP	5600	<0.01				10187607 EPA8310	251020 HPLC-UV	Acceptable
Chrysene	X	WP	5855	<0.1				10107401 EPA625	151010 GC-MS	Acceptable
Chrysene	X	WP	5855	<0.01				10187607 EPA8310	251020 HPLC-UV	Acceptable
Dibenzo(a,h)anthracene	X	WP	5895	<0.1				10107401 EPA625	151010 GC-MS	Acceptable
Dibenzo(a,h)anthracene	X	WP	5895	<0.05				10187607 EPA8310	251020 HPLC-UV	Acceptable
Fluoranthene	X	WP	6265	0.852	1.53	0.540-1.80 0.748-1.59	1.51	10187607 EPA8310	251020 HPLC-UV	Acceptable
Fluoranthene	X	WP	6265	0.68	1.53	0.540-1.80 0.748-1.59	2.32	10107401 EPA625	151010 GC-MS	**Check for Error**
Fluorene	X	WP	6270	2.53	5.56	1.35-6.83 2.26-5.92	1.71	10107401 EPA625	151010 GC-MS	Acceptable
Fluorene	X	WP	6270	2.31	5.56	1.35-6.83 2.26-5.92	1.95	10187607 EPA8310	251020 HPLC-UV	Acceptable

Performance Summary

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APG Customer 3655	Laboratory Address	Nelac Point Of Contact	WP October 2004 WP #4010
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date 11/23/2004
Primary AA E-10254	15130 South Keeler	QA Director	Study Shipment Date 10/01/2004
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date 11/15/2004

Product: Polynuclear Aromatic Hydrocarbons-PAH **WP Lot Number:** 40813 **APG+ Lot Number:** 40814 **Units:** ug/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Indeno(1,2,3-cd)pyrene	X	WP	6315	<0.1				10107401 EPA625	151010 GC-MS	Acceptable
Indeno(1,2,3-cd)pyrene	X	WP	6315	<0.01				10187607 EPA8310	251020 HPLC-UV	Acceptable
Naphthalene	X	WP	5005	6.54	15.3	1.30-19.3 4.28-16.3	1.25	10107401 EPA625	151010 GC-MS	Acceptable
Naphthalene	X	WP	5005	4.21	15.3	1.30-19.3 4.28-16.3	2.02	10187607 EPA8310	251020 HPLC-UV	**Check for Error**
Phenanthrene	X	WP	6615	0.622	1.24	0.346-1.64 0.562-1.43	1.72	10187607 EPA8310	251020 HPLC-UV	Acceptable
Phenanthrene	X	WP	6615	0.56	1.24	0.346-1.64 0.562-1.43	2.01	10107401 EPA625	151010 GC-MS	**Check for Error**
Pyrene	X	WP	6665	0.628	1.04	0.539-1.14 0.640-1.04	2.12	10187607 EPA8310	251020 HPLC-UV	**Check for Error**
Pyrene	X	WP	6665	0.50	1.04	0.539-1.14 0.640-1.04	3.40	10107401 EPA625	151010 GC-MS	**Not Acceptable**

Product: Solids **WP Lot Number:** 94756 **Units:** mg/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Dissolved Solids		WP	1705	278	309	234-389 260-363	1.32	10009004 EPA160.1	201010 GRAV	Acceptable
Total Solids		WP	1950	340	354	259-461 292-427	0.593	10009800 EPA160.3	201010 GRAV	Acceptable
Total Suspended Solids	NE	WP	1960	42	45.1	33.8-48.3 36.2-45.9	0.413	10009402 EPA160.2	201010 GRAV	Acceptable

Performance Summary

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APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP October 2004 WP #4010
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/23/2004
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2004
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/15/2004

Product: Total Chlordane WP Lot Number: 40892 APG+ Lot Number: 40893 Units: ug/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Chlordane	NE	WP	7250	3.36	3.54	1.55-5.03	0.121	10103603	152010	Acceptable
						2.13-4.45		EPA608	GC-ECD	

Product: Total Cyanide WP Lot Number: 40762 APG+ Lot Number: 40763 Units: mg/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Cyanide	NE	WP	1645	0.097	0.0940	0.0578-0.128	0.371	10060205	401010	Acceptable
						0.0695-0.116		EPA335.2	COLOR	

Product: Total Phenolics WP Lot Number: 40754 APG+ Lot Number: 40755 Units: mg/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Phenolics	NE	WP	1905	0.209	0.418	0.145-0.408	1.55	10079604	401010	Acceptable
						0.189-0.365		EPA420.2	COLOR	

Product: Total Residual Chlorine WP Lot Number: 40917 APG+ Lot Number: 40918 Units: mg/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Residual Chlorine	NE	WP	1940	1.15	1.11	0.814-1.29	1.27	10059004	651010	Acceptable
						0.893-1.21		EPA330.4	TITR	

Product: Toxaphene WP Lot Number: 40894 APG+ Lot Number: 40895 Units: ug/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Toxaphene	N	WP	8250	3.90	4.28	0.834-7.50	0.243	10103603	152010	Acceptable
						1.95-6.39		EPA608	GC-ECD	

Performance Summary

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APG Customer	3655	Laboratory Address	Nelac Point Of Contact	WP October 2004 WP #4010	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/23/2004
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2004
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/15/2004

Product: TPH Diesel (DRO) **WP Lot Number: 40861** **APG+ Lot Number: 40862** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
TPH Diesel (DRO)	X	WP	9369	1.23	3.10	0.546-3.61	1.66	10173203	152510	Acceptable
						1.06-3.10		EPA8015	GC-FID	

Product: TPH Gasoline (GRO) **WP Lot Number: 40863** **APG+ Lot Number: 40864** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
TPH Gasoline (GRO)	X	WP	9408	0.12	0.174	0.0391-0.302	1.16	10173203	152510	Acceptable
						0.0834-0.259		EPA8015	GC-FID	

Performance Summary

3655

APG Customer	3655	Laboratory Address	Nelac Point Of Contact	WP October 2004 WP #4010	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/23/2004
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2004
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/15/2004

Product: Trace Metals **WP Lot Number: 40876-40877** **APG+ Lot Number: 40878-40898** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aluminum	NE	WP	1000	1740	1740	1490-1980 1570-1890	0.124	10013408 EPA200.7	102015 ICP-AES	Acceptable
Aluminum	NE	WP	1000	1810	1740	1490-1980 1570-1890	0.995	10014401 EPA200.8	102010 ICP-MS	Acceptable
Antimony	NE	WP	1005	666	700	494-841 552-784	0.0346	10013408 EPA200.7	102015 ICP-AES	Acceptable
Antimony	NE	WP	1005	695	700	494-841 552-784	0.467	10014401 EPA200.8	102010 ICP-MS	Acceptable
Arsenic	NE	WP	1010	423	407	340-478 363-455	0.611	10013408 EPA200.7	102015 ICP-AES	Acceptable
Arsenic	NE	WP	1010	402	407	340-478 363-455	0.306	10014401 EPA200.8	102010 ICP-MS	Acceptable
Barium	N	WP	1015	259	263	230-303 243-291	0.661	10013408 EPA200.7	102015 ICP-AES	Acceptable
Barium	N	WP	1015	248	263	230-303 243-291	1.57	10014401 EPA200.8	102010 ICP-MS	Acceptable
Beryllium	NE	WP	1020	339	362	307-409 324-392	1.12	10013408 EPA200.7	102015 ICP-AES	Acceptable
Beryllium	NE	WP	1020	344	362	307-409 324-392	0.828	10014401 EPA200.8	102010 ICP-MS	Acceptable
Boron	X	WP	1025		624	520-735 556-700				Not Reported
Boron	X	WP	1025	640	624	520-735 556-700	0.334	10014401 EPA200.8	102010 ICP-MS	Acceptable
Cadmium	NE	WP	1030	109	102	86.3-117 91.8-112	1.37	10013408 EPA200.7	102015 ICP-AES	Acceptable

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP October 2004 WP #4010
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/23/2004
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2004
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/15/2004

Product: Trace Metals **WP Lot Number: 40876-40877** **APG+ Lot Number: 40878-40898** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Cadmium	NE	WP	1030	101	102	86.3-117 91.8-112	0.196	10014401 EPA200.8	102010 ICP-MS	Acceptable
Chromium	NE	WP	1040	786	753	657-851 689-819	0.988	10013408 EPA200.7	102015 ICP-AES	Acceptable
Chromium	NE	WP	1040	757	753	657-851 689-819	0.0926	10014401 EPA200.8	102010 ICP-MS	Acceptable
Cobalt	NE	WP	1050	126	123	107-139 112-134	0.561	10013408 EPA200.7	102015 ICP-AES	Acceptable
Cobalt	NE	WP	1050	121	123	107-139 112-134	0.374	10014401 EPA200.8	102010 ICP-MS	Acceptable
Copper	NE	WP	1055	606	589	535-647 554-628	0.802	10013408 EPA200.7	102015 ICP-AES	Acceptable
Copper	NE	WP	1055	574	589	535-647 554-628	0.909	10014401 EPA200.8	102010 ICP-MS	Acceptable
Iron	NE	WP	1070	2410	2330	2070-2620 2150-2530	0.753	10013408 EPA200.7	102015 ICP-AES	Acceptable
Iron	NE	WP	1070	2260	2330	2070-2620 2150-2530	0.861	10014401 EPA200.8	102010 ICP-MS	Acceptable
Lead	NE	WP	1075	1190	1190	1050-1330 1100-1280	0.000	10013408 EPA200.7	102015 ICP-AES	Acceptable
Lead	NE	WP	1075	1210	1190	1050-1330 1100-1280	0.422	10014401 EPA200.8	102010 ICP-MS	Acceptable
Manganese	NE	WP	1090	261	251	224-279 233-269	1.09	10013408 EPA200.7	102015 ICP-AES	Acceptable
Manganese	NE	WP	1090	241	251	224-279 233-269	1.09	10014401 EPA200.8	102010 ICP-MS	Acceptable

Performance Summary

3655

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP October 2004 WP #4010
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	11/23/2004
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/01/2004
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/15/2004

Product: Trace Metals **WP Lot Number: 40876-40877** **APG+ Lot Number: 40878-40898** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Mercury	NE	WP	1095	16.9	16.4	12.3-20.4 13.7-19.1	0.370	10036201 EPA245.1	101010 CVAAS	Acceptable
Mercury	NE	WP	1095	14.5	16.4	12.3-20.4 13.7-19.1	1.41	10014401 EPA200.8	102010 ICP-MS	Acceptable
Molybdenum	NE	WP	1100	181	182	154-209 164-200	0.110	10013408 EPA200.7	102015 ICP-AES	Acceptable
Molybdenum	NE	WP	1100	176	182	154-209 164-200	0.659	10014401 EPA200.8	102010 ICP-MS	Acceptable
Nickel	NE	WP	1105	818	806	728-901 756-872	0.138	10013408 EPA200.7	102015 ICP-AES	Acceptable
Nickel	NE	WP	1105	797	806	728-901 756-872	0.588	10014401 EPA200.8	102010 ICP-MS	Acceptable
Selenium	NE	WP	1140	784	807	641-934 689-885	0.0612	10013408 EPA200.7	102015 ICP-AES	Acceptable
Selenium	NE	WP	1140	778	807	641-934 689-885	0.184	10014401 EPA200.8	102010 ICP-MS	Acceptable
Silver	NE	WP	1150	76.6	69.9	59.4-80.3 62.8-76.8	1.94	10013408 EPA200.7	102015 ICP-AES	Acceptable
Silver	NE	WP	1150	64.5	69.9	59.4-80.3 62.8-76.8	1.51	10014401 EPA200.8	102010 ICP-MS	Acceptable
Strontium	NE	WP	1160		211	179-242 190-232				Not Reported
Strontium	NE	WP	1160	197	211	179-242 190-232	1.35	10014401 EPA200.8	102010 ICP-MS	Acceptable
Thallium	NE	WP	1165	671	678	547-786 587-747	0.100	10013408 EPA200.7	102015 ICP-AES	Acceptable

Performance Summary

3655

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		**WP October 2004 WP #4010**
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**11/23/2004**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**10/01/2004**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**11/15/2004**

Product: Trace Metals **WP Lot Number: 40876-40877** **APG+ Lot Number: 40878-40898** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Thallium	NE	WP	1165	640	678	547-786 587-747	0.677	10014401 EPA200.8	102010 ICP-MS	Acceptable
Tin	N	WP	1175		1300	1030-1570 1120-1480				Not Reported
Tin	N	WP	1175	1260	1300	1030-1570 1120-1480	0.442	10014401 EPA200.8	102010 ICP-MS	Acceptable
Titanium	NE	WP	1180		145	124-164 131-157				Not Reported
Titanium	NE	WP	1180	131	145	124-164 131-157	1.95	10014401 EPA200.8	102010 ICP-MS	Acceptable
Vanadium	NE	WP	1185	5060	5040	4550-5540 4720-5380	0.0610	10013408 EPA200.7	102015 ICP-AES	Acceptable
Vanadium	NE	WP	1185	5010	5040	4550-5540 4720-5380	0.244	10014401 EPA200.8	102010 ICP-MS	Acceptable
Zinc	NE	WP	1190	770	724	641-815 670-786	1.45	10013408 EPA200.7	102015 ICP-AES	Acceptable
Zinc	NE	WP	1190	714	724	641-815 670-786	0.483	10014401 EPA200.8	102010 ICP-MS	Acceptable

Performance Summary

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APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP October 2004 WP #4010
Study Print Date 11/23/2004
Study Shipment Date 10/01/2004
Study Closing Date 11/15/2004

Product: Volatiles **WP Lot Number: 40808** **APG+ Lot Number: 40809** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
1,1,1-Trichloroethane	NE	WP	5160	37.5	34.5	22.4-45.5 26.3-41.7	0.909	10107207 EPA624	151010 GC-MS	Acceptable
1,1,2,2-Tetrachloroethane	N	WP	5110	42.3	36.4	20.9-51.0 26.0-46.0	1.25	10107207 EPA624	151010 GC-MS	Acceptable
1,1,2-Trichloroethane	N	WP	5165	59.2	54.4	37.3-70.6 42.9-65.1	0.939	10107207 EPA624	151010 GC-MS	Acceptable
1,1-Dichloroethane	X	WP	4630	<2				10107207 EPA624	151010 GC-MS	Acceptable
1,1-Dichloroethene	N	WP	4640	127	123	69.2-185 88.4-166	0.000	10107207 EPA624	151010 GC-MS	Acceptable
1,2-Dichloroethane	NE	WP	4635	32.6	29.7	20.6-39.9 23.9-36.7	0.717	10107207 EPA624	151010 GC-MS	Acceptable
1,2-Dichloropropane	N	WP	4655	144	136	91.1-173 105-159	0.876	10107207 EPA624	151010 GC-MS	Acceptable
Bromodichloromethane	NE	WP	4395	65.8	60.7	43.1-78.9 49.1-72.9	0.804	10107207 EPA624	151010 GC-MS	Acceptable
Bromoform	NE	WP	4400	58.2	65.1	42.7-89.0 50.5-81.3	0.997	10107207 EPA624	151010 GC-MS	Acceptable
Bromomethane	N	WP	4950	<2				10107207 EPA624	151010 GC-MS	Acceptable
Carbon tetrachloride	NE	WP	4455	48.8	45.6	28.2-64.3 34.3-58.3	0.415	10107207 EPA624	151010 GC-MS	Acceptable
Chlorobenzene	NE	WP	4475	14.0	13.6	10.2-17.1 11.3-15.9	0.345	10107207 EPA624	151010 GC-MS	Acceptable
Chloroethane	N	WP	4485	<2				10107207 EPA624	151010 GC-MS	Acceptable

Performance Summary

3655

APG Customer 3655	Laboratory Address	Nelac Point Of Contact	**WP October 2004 WP #4010**
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date 11/23/2004**
Primary AA E-10254	15130 South Keeler	QA Director	**Study Shipment Date 10/01/2004**
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date 11/15/2004**

Product: Volatiles **WP Lot Number: 40808** **APG+ Lot Number: 40809** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Chloroform	NE	WP	4505	49.4	45.8	31.8-58.8	0.913	10107207	151010	Acceptable
						36.3-54.3		EPA624	GC-MS	
Chloromethane	N	WP	4960	<2				10107207	151010	Acceptable
								EPA624	GC-MS	
Dibromochloromethane	NE	WP	4575	20.2	19.0	12.5-25.2	0.664	10107207	151010	Acceptable
						14.6-23.0		EPA624	GC-MS	
Methylene chloride	NE	WP	4975	26.2	23.7	15.0-33.2	0.693	10107207	151010	Acceptable
						18.0-30.2		EPA624	GC-MS	
Tetrachloroethene	NE	WP	5115	30.0	32.7	21.6-42.1	0.556	10107207	151010	Acceptable
						25.1-38.7		EPA624	GC-MS	
trans-1,2-Dichloroethene	N	WP	4700	120	129	82.5-176	0.577	10107207	151010	Acceptable
						97.8-160		EPA624	GC-MS	
trans-1,3-Dichloropropene	N	WP	4685	<2				10107207	151010	Acceptable
								EPA624	GC-MS	
Trichloroethene	NE	WP	5170	69.1	71.8	46.5-92.7	0.0649	10107207	151010	Acceptable
						54.2-85.0		EPA624	GC-MS	
Trichlorofluoromethane	N	WP	5175	<2				10107207	151010	Acceptable
								EPA624	GC-MS	
Vinyl Chloride	N	WP	5235	<2				10107207	151010	Acceptable
								EPA624	GC-MS	

Analytical Products Group, Inc.

PERFORMANCE REPORT

WP Performance Summary

April 2005 WP

APG Customer Code: 3655

Analytical Management Lab
Suite B
15130 South Keeler
Olathe, KS 66062

Laboratory of
EXCELLENCE

NVLAP®
LAB CODE 200384-0

Study Certification
April 2005 WP

I have reviewed the statistical analysis 271 which was performed on April 2005 WP. The statistical analysis yielded results which are consistent with NIST and NELAC Requirements.

Thomas V. Coyner
Technical Director

I have reviewed these results and find them to meet the appropriate NELAC and NIST Requirements.

Quality Assurance Manager

This report was produced at
Analytical Products Group, Inc
2730 Washington Blvd.
Belpre, Ohio 45714

This report has been produced in accordance with Analytical Products Group, Inc. ISO 9001:2000 Certification (CERT-9513-2003-AQ-HOU-RAB) issued by Det Norske Vertitas.

Water Pollution (WP) Proficiency Testing Program Report

Introduction:

The Analytical Products Group, Inc. Proficiency Testing Program has been designed to evaluate your laboratory's performance against other participating laboratories on the same set of standards. This report has been specifically prepared for your organization to assist you in ensuring that your procedures are in-control. Therefore, in most cases sample levels are not designed to challenge the detection limits of the method.

The Analytical Products Group Proficiency Testing Program is certified to meet all of the following requirements:

- For applicable analytes, this Proficiency Testing Program is accredited by NIST according to the National Voluntary Laboratory Accreditation Program (Handbook 150-19) for Chemical Calibrations: Providers of Proficiency Testing (NVLAP Code 200384-0)

- Analytical Products Group, Inc. Quality System is registered with DNVCertification to meet the ISO 9001 standard to design, manufacture, and distribute chemical reference materials and operate Proficiency Testing Programs (Certificate # 98-HOU-AQ-9513)

- For applicable analytes, this Proficiency Testing Program is certified to meet all requirements as specified in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).

Please note that this report contains data that are not covered by the NVLAP accredition. This report must not be used to claim product certification, approval, or endorsement by NVLAP, NIST, or any agency of the federal government.

Report Layout

This report contains the following sub-reports:

Introduction -This section is the general introduction to the Analytical Products Group, Inc. Proficiency Testing Program as well as all of the certifications that the organization maintains.

Terms & Definitions - Defines all of the terms used throughout the report. This is an excellent section to refer to first if you have any questions.

Laboratory Benchmark Report - This report presents an overall summary of the study as well as your performance relative to all other study participants.

Comment Report - Contains comments submitted to Analytical Products Group by the program participants or comments by Analytical Products Group to program participants concerning unusual characteristics of the data set or problems with standards which could influence the interpretation of the data. Please review this section thoroughly before examining your data.

Exception Report - This report is a summary of all of the analytes that resulted in an Evaluation of "Not Acceptable" for your laboratory. The Exception Report is designed to very quickly identify for your laboratory the areas that may require your immediate attention.

Performance Evaluation Summary Report - Provides a summary of all of your data for each analyte. This summary is formatted similar to the US EPA DMR-QA Reports.

Detail Report- This report lists all the details of your data. Also included in this section are useful graphs indicating the APG Assigned Value, the Mean, the Acceptance Limits and your Reported Value. Another graph included in this section shows your Z score for the analyte of interest.

The goal of this report is to provide your laboratory with the information you need to monitor and improve your quality. If you have any questions or feedback, please give us a call (800-272-4442). Thank you for choosing Analytical Products Group as your proficiency testing provider.

Sincerely,

Gregg Frazier
General Manager

Terms & Definitions

Average Acceptable Data - This value is the average percent of data that is within the EPA Acceptance Limits for this study. The Average Acceptable Data is reported for all laboratories as a whole as well as your laboratory as an individual. These values can be found in the Laboratory Benchmark Report.

Average Percentile for All Parameters - This is your laboratory's rank on each parameter and the related rank for that parameter. The average percentile is the average of this data across all parameters reported. It is a different measure of performance than percent acceptable data since it is affected by your relative performance on each parameter. The report provides an average for this measure for all laboratories reporting as well as your laboratory's performance.

Analyte - Name of the analyte of interest as found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) or NELAC Field of Testing List.

NELAC Analyte Code - This value is the code given to the analyte by NELAC. These codes can be found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) or the NELAC Fields of Testing List.

APG Assigned Value - This value is the calculated True Value of the standard based upon the actual composition of the standard in most cases.

Z-Score - This figure corresponds to how far your Reported Value was from the EPA Mean, reported in terms of EPA Standard Deviation units. As a point of reference, the US EPA Acceptance Limits are plus or minus three (3) EPA Standard Deviations from the EPA Mean.

APG Certified Value - This value is the result of the quality control testing performed on the sample by Analytical Products Group before the sample is shipped to the customer. Through this testing, the samples are traced against a NIST Standard Reference Material where available or an external standard if a NIST Standard Reference Material is not available. The reported uncertainty of this value is expressed as two times the standard deviation (s) divided by the square root of the number of samples (n) tested (i.e. 2s/vn). This quality control testing meets the requirements as specified by the NIST National Voluntary Laboratory Accreditation Program (NVLAP) Handbook 150-19 (Chemical Calibration: Providers of Proficiency Testing).

Acceptance Range - For applicable analytes, this range is calculated based upon the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045). For the Water Pollution Program (WP), EPA Acceptance Limits are defined as ± three (3) EPA Standard Deviations from the EPA Mean.

Mean - If the analyte is included in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then this value is the estimated mean value calculated per the Criteria Document. If the analyte is not included in the Criteria Document, then this value is the Study Mean.

Study Standard Deviation - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Standard Deviation is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Your Ranking - This is your ranking based on your Z Score for the analyte of interest among all possible rankings. It is important to note that several laboratories can achieve the same rank.

Terms & Definitions (continued)

Evaluation - This value is determined by the Acceptance Limits and the Reported Value. Evaluation may have one of following values:

Value	Description
Acceptable	The Reported Value was within the Acceptable Limits or a less than (<) value was reported AND zero (0) was included in the Acceptance Limit Range AND the numeric part of the Reported Value was less than the upper limit of the Acceptance Limit.
Not Acceptable	The Reported Value is outside of the Acceptance Limits or a less than (<) value was reported AND zero (0) is not included in the Acceptance Limits range or the numeric part of the Reported Value was greater than the upper limit of the Acceptance Limit or a greater than value (>) was reported.
Check for Error	The Reported Value is between two (2) and three (3) Standard Deviations from the Mean.
Not Evaluated	The Reported Value was a text value which could not be evaluated.
Blank	No data was reported for this analyte [the analyte was not spiked].

Inter-Laboratory Statistics - This data is a summary of all of the data from all study participants for the analyte of interest. Inter-Laboratory Statistics are only reported if a minimum number of laboratories analyzed and reported data for the analyte of interest.

Product Level - Often referred to as 'Program'. This value indicates which program the analyte of interest is a part. For Water Pollution Studies (WP), the only values that Program will have are 'WP' or 'APG+'. If the analyte and range are consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then the Program value will have a value of 'WP' and if this is not the case then the Program value will be 'APG+', indicating that the analyte and/or range was specified by Analytical Products Group.

Product Type - To the right of each analyte in the performance summary is a code that designates the scope of the APG's accreditation for that analyte. An analyte designated as "NE" is applicable to NELAC and EPA requirements and ranges. An analyte designated as "N" is applicable to NELAC requirements and ranges. An analyte designated as "E" is applicable to EPA requirements and ranges. An analyte designated as "X" or without a designation is not covered by APG's accreditations.

Rank on Percent Acceptable Data - This is your laboratories rank out of all possible ranks in the current study based on percent acceptable data. It is important to note that several laboratories can achieve the same rank.

Rank on Average Percentile Data - This value is your laboratories rank out of all possible ranks based on Your Ranking converted to a percentile. It is important to note that several laboratories can achieve the same rank.

Laboratory Pass Percentage - This is the percentage of study participants that reported values within the Acceptance Limits for the analyte of interest.

Solids Standards - Solids standards are individually prepared, therefore, each standard has a unique APG Assigned Value.

Terms & Definitions (continued)

Reported Value - This value is the result that was reported by your organization for the particular analyte. Every effort has been made to ensure correct data entry. Should you find a data entry error, please notify Analytical Products Group, Inc. and we will correct the error and re-issue the report. If the Reported Value is reported as a less than (<) figure, then the only way that the Evaluation will result in an "Acceptable" value is if the numeric part of the Reported Value (i.e. 8 if the Report Value were <8) is less than the upper limit of the Acceptance Range AND zero (0) is included in the Acceptance Range. Any other non-numeric value (i.e. BDL, ND, >10, etc.) cannot be evaluated and will return an Evaluation result of "No Evaluation". This method of evaluation is consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).

Number of Acceptable Data Points - This value is the number of data points reported by your laboratory that were within the Acceptance Limits.

Weighted Rank on Percent Acceptable Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percent Acceptable Data is the weighted rank of your laboratory based on Percent Acceptable Data and the number of analytes reported.

Weight Rank on Percentile Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percentile Data is the weighted rank of your laboratory based on Rank on Average Percentile Data and the number of analytes reported.

Study Mean - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Mean is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Laboratory Benchmark Report

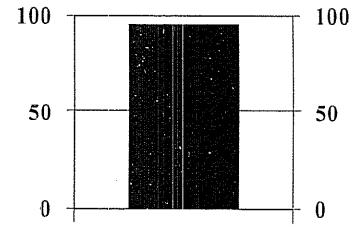
Customer Code: 3655 **April 2005 WP**

Summary of Ranking Data

Summary of National Ranking Data

Total Number of Laboratories Reporting:	307
Average Percent Acceptable Data:	95.83%
Average Percentile for all Parameters:	54.69%

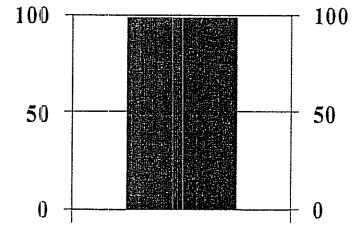
National Avg % Acceptable
95.8 %



Summary of Laboratory Ranking:

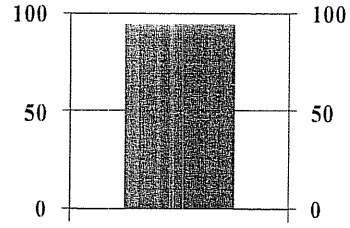
Total Data Points Reported:	228
Number of Acceptable Data Points:	225
Percent Acceptable Data:	98.68%
Average Percentile for all Parameters:	54.09%

Laboratory Avg % Acceptable
98.68%



Rank on Percent Acceptable Data:	14 of 100
Rank on Average Percentile Data:	149 of 258
Weighted Rank on Percent Acceptable Data:	15 of 145
Weighted Rank on Average Percentile Data:	14 of 282

Weighted Rank Avg Percentile
95.0 %



Comment Report

There are no comments to report.

Exception Report

Product: Demand — **Exception Reporting** — **Units: mg/L**

Analyte		Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code / Method Desc.	Technology Code / Technology Desc.	Evaluation
Total Organic Carbon	NE	WP	2040	16.7	32.5	27.1-37.6	8.92	10078203 EPA415.1	402010 IR	Not Acceptable

Product: Minerals — **Exception Reporting** — **Units: umhos/cm**

Analyte		Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code / Method Desc.	Technology Code / Technology Desc.	Evaluation
Conductivity	NE	WP	1610	293	331	298-346	3.56	10006209 EPA120.1	351010 COND	Not Acceptable

Product: Solids — **Exception Reporting** — **Units: mg/L**

Analyte		Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code / Method Desc.	Technology Code / Technology Desc.	Evaluation
Total Solids		WP	1950	75.0	109	81.9-137	3.70	10009800 EPA160.3	201010 GRAV	Not Acceptable

Performance Summary

Performance Summary

APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

3655
WP April 2005 WP #5004
Study Print Date 06/06/2005
Study Shipment Date 04/04/2005
Study Closing Date 05/18/2005

Product: Acid Extractables **WP Lot Number: 42584** **APG+ Lot Number: 42585** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
2,4,5-Trichlorophenol	N	WP	6835	73.6	109	41.7-138 57.8-122	1.02	10107401 EPA625	151010 GC-MS	Acceptable
2,4,6-Trichlorophenol	N	WP	6840	48.0	67.2	22.9-88.4 33.8-77.4	0.697	10107401 EPA625	151010 GC-MS	Acceptable
2,4-Dichlorophenol	N	WP	6000	36.0	51.9	18.8-64.5 26.4-56.8	0.736	10107401 EPA625	151010 GC-MS	Acceptable
2,4-Dimethylphenol	N	WP	6130	73.6	124	26.8-163 49.5-140	0.934	10107401 EPA625	151010 GC-MS	Acceptable
2,4-Dinitrophenol	N	WP	6175	<10				10107401 EPA625	151010 GC-MS	Acceptable
2-Chlorophenol	N	WP	5800	83.4	173	48.6-217 77.0-189	1.77	10107401 EPA625	151010 GC-MS	Acceptable
2-Methyl-4,6-Dinitrophenol	N	WP	6360	<10				10107401 EPA625	151010 GC-MS	Acceptable
2-Nitrophenol	N	WP	6490	55.2	85.6	25.9-112 40.2-97.8	0.958	10107401 EPA625	151010 GC-MS	Acceptable
4-Chloro-3-methylphenol	N	WP	5700	76.0	107	41.5-138 57.5-122	0.844	10107401 EPA625	151010 GC-MS	Acceptable
4-Nitrophenol	N	WP	6500	98.3	150	0.000-202 3.40-162	0.396	10107401 EPA625	151010 GC-MS	Acceptable
Pentachlorophenol	N	WP	6605	36.1	38.5	5.95-53.2 13.9-45.3	0.826	10107401 EPA625	151010 GC-MS	Acceptable
Phenol	N	WP	6625	68.3	135	0.000-181 5.40-146	0.213	10107401 EPA625	151010 GC-MS	Acceptable

Performance Summary

APG Customer 3655
EPA Lab Code KS00902
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APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP April 2005 WP #5004
Study Print Date 06/06/2005
Study Shipment Date 04/04/2005
Study Closing Date 05/18/2005

Product: Aluminum High Level **WP Lot Number: 42484** **APG+ Lot Number: 42485** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aluminum High Level	X	WP	1000	0.144	0.201	0.0984-0.264 0.126-0.236	1.34	10013408 EPA200.7	102015 ICP-AES	Acceptable

Performance Summary

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APG Customer	3655	Laboratory Address	Nelac Point Of Contact		**WP April 2005 WP #5004**
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**06/06/2005**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**04/04/2005**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**05/18/2005**

Product: Base Neutral Extractables **WP Lot Number: 42595** **APG+ Lot Number: 42596** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
1,2,4-Trichlorobenzene	N	WP	5155	<10				10107401 EPA625	151010 GC-MS	Acceptable
1,2-Dichlorobenzene		WP	4610	51.5	95.7	25.9-98.7 38.1-86.5	0.893	10107401 EPA625	151010 GC-MS	Acceptable
1,3-Dichlorobenzene		WP	4615	<10				10107401 EPA625	151010 GC-MS	Acceptable
1,4-Dichlorobenzene		WP	4620	30.3	48.5	15.0-47.2 20.4-41.8	0.149	10107401 EPA625	151010 GC-MS	Acceptable
2,4-Dinitrotoluene (2,4-DNT)	N	WP	6185	97.9	140	59.3-187 80.6-165	1.18	10107401 EPA625	151010 GC-MS	Acceptable
2,6-Dinitrotoluene (2,6-DNT)	N	WP	6190	27.3	36.0	13.8-44.7 18.9-39.5	0.369	10107401 EPA625	151010 GC-MS	Acceptable
2-Chloronaphthalene	N	WP	5795	52.8	89.6	35.7-103 47.0-91.8	1.48	10107401 EPA625	151010 GC-MS	Acceptable
3,3'-Dichlorobenzidine	N	WP	5945	<10				10107401 EPA625	151010 GC-MS	Acceptable
4-Bromophenyl phenyl ether	N	WP	5660	<10				10107401 EPA625	151010 GC-MS	Acceptable
4-Chlorophenyl phenyl ether	N	WP	5825	<10				10107401 EPA625	151010 GC-MS	Acceptable
Acenaphthene	N	WP	5500	77.8	154	56.8-197 80.4-174	2.11	10107401 EPA625	151010 GC-MS	**Check for Error**
Acenaphthylene	N	WP	5505	21.7	27.8	10.2-35.6 14.5-31.3	0.284	10107401 EPA625	151010 GC-MS	Acceptable
Anthracene	N	WP	5555	54	114	39.8-147 57.7-129	2.21	10107401 EPA625	151010 GC-MS	**Check for Error**

Performance Summary

3655

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP April 2005 WP #5004
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	06/06/2005
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	04/04/2005
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	05/18/2005

Product: Base Neutral Extractables **WP Lot Number: 42595** **APG+ Lot Number: 42596** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Benzidine	N	WP	5595	<10				10107401 EPA625	151010 GC-MS	Acceptable
Benzo(a)anthracene	N	WP	5575	32.8	39.0	22.1-45.7 26.0-41.8	0.280	10107401 EPA625	151010 GC-MS	Acceptable
Benzo(a)pyrene	N	WP	5580	34.1	46.9	9.52-61.7 18.2-53.0	0.172	10107401 EPA625	151010 GC-MS	Acceptable
Benzo(b)fluoranthene	N	WP	5585	<10				10107401 EPA625	151010 GC-MS	Acceptable
Benzo(g,h,i)perylene	N	WP	5590	<10				10107401 EPA625	151010 GC-MS	Acceptable
Benzo(k)fluoranthene	N	WP	5600	<10				10107401 EPA625	151010 GC-MS	Acceptable
Bis(2-Chloroethoxy) methane	N	WP	5760	<10				10107401 EPA625	151010 GC-MS	Acceptable
Bis(2-Chloroethyl)ether	N	WP	5765	<10				10107401 EPA625	151010 GC-MS	Acceptable
Bis(2-Chloroisopropyl) ether	N	WP	5780	<10				10107401 EPA625	151010 GC-MS	Acceptable
Bis(2-Ethylhexyl) phthalate	N	WP	6255	62.1	108	19.0-158 42.3-135	1.14	10107401 EPA625	151010 GC-MS	Acceptable
Butyl benzyl phthalate	N	WP	5670	35.3	43.0	4.29-69.3 15.2-58.4	0.139	10107401 EPA625	151010 GC-MS	Acceptable
Chrysene	N	WP	5855	33.4	41.2	18.1-53.2 24.0-47.4	0.393	10107401 EPA625	151010 GC-MS	Acceptable
Di-n-butyl phthalate	N	WP	5925	39.2	66.5	14.6-94.3 27.9-81.1	1.15	10107401 EPA625	151010 GC-MS	Acceptable

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		**WP April 2005 WP #5004**
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**06/06/2005**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**04/04/2005**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**05/18/2005**

Product: Base Neutral Extractables **WP Lot Number: 42595** **APG+ Lot Number: 42596** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Di-n-octyl phthalate	N	WP	6200	38.0	61.2	11.6-90.5 24.9-77.3	1.00	10107401 EPA625	151010 GC-MS	Acceptable
Dibenzo(a,h)anthracene	N	WP	5895	<10				10107401 EPA625	151010 GC-MS	Acceptable
Diethyl phthalate	N	WP	6070	86.8	167	0.000-256 29.2-211	0.731	10107401 EPA625	151010 GC-MS	Acceptable
Dimethyl phthalate	N	WP	6135	93.2	166	0.000-254 2.20-204	0.194	10107401 EPA625	151010 GC-MS	Acceptable
Fluoranthene	N	WP	6265	18.2	24.0	13.0-30.4 15.9-27.5	1.20	10107401 EPA625	151010 GC-MS	Acceptable
Fluorene	N	WP	6270	20.9	32.6	13.9-42.7 18.7-37.9	1.54	10107401 EPA625	151010 GC-MS	Acceptable
Hexachlorobenzene	N	WP	6275	17.6	24.2	10.5-31.6 14.0-28.2	0.992	10107401 EPA625	151010 GC-MS	Acceptable
Hexachlorobutadiene	N	WP	4835	63.1	140	24.5-167 48.4-144	1.38	10107401 EPA625	151010 GC-MS	Acceptable
Hexachlorocyclopentadiene	N	WP	6285	32.8	47.9	0.000-64.0 4.10-52.1	0.392	10107401 EPA625	151010 GC-MS	Acceptable
Hexachloroethane	N	WP	4840	67.5	122	10.4-155 34.4-131	0.627	10107401 EPA625	151010 GC-MS	Acceptable
Indeno (1,2,3-cd) pyrene	N	WP	6315	<10				10107401 EPA625	151010 GC-MS	Acceptable
Isophorone	N	WP	6320	40.1	54.9	20.9-72.1 29.4-63.6	0.750	10107401 EPA625	151010 GC-MS	Acceptable
N-nitrosodi-n-propylamine	N	WP	6545	<10				10107401 EPA625	151010 GC-MS	Acceptable

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact	**WP April 2005 WP #5004**	
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**06/06/2005**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**04/04/2005**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**05/18/2005**

Product: Base Neutral Extractables **WP Lot Number: 42595** **APG+ Lot Number: 42596** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range / Warn Range	ZScore	Method Code / Method Desc.	Technology Code / Technology Desc.	Evaluation
N-nitrosodimethylamine	N	WP	6530	<10				10107401 EPA625	151010 GC-MS	Acceptable
N-nitrosodiphenylamine	N	WP	6535	<10				10107401 EPA625	151010 GC-MS	Acceptable
Naphthalene	N	WP	5005	42.2	96.2	24.7-126 / 41.5-109	1.96	10107401 EPA625	151010 GC-MS	Acceptable
Nitrobenzene (NB)	N	WP	5015	74.2	146	46.7-191 / 71.0-167	1.87	10107401 EPA625	151010 GC-MS	Acceptable
Phenanthrene	N	WP	6615	39.5	68.0	29.2-87.4 / 38.9-77.7	1.94	10107401 EPA625	151010 GC-MS	Acceptable
Pyrene	N	WP	6665	73.6	135	44.9-182 / 67.4-159	1.73	10107401 EPA625	151010 GC-MS	Acceptable

Performance Summary

3655

APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP April 2005 WP #5004
Study Print Date 06/06/2005
Study Shipment Date 04/04/2005
Study Closing Date 05/18/2005

Product: BTEX **WP Lot Number: 42508** **APG+ Lot Number: 42509** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range / Warn Range	ZScore	Method Code / Method Desc.	Technology Code / Technology Desc.	Evaluation
Benzene	NE	WP	4375	57.0	70.4	50.9-90.4 / 57.5-83.7	2.07	10107207 / EPA624	151010 / GC-MS	**Check for Error**
Ethylbenzene	NE	WP	4765	25.2	29.8	20.0-38.8 / 23.1-35.7	1.34	10107207 / EPA624	151010 / GC-MS	Acceptable
m-Xylene / p-Xylene		WP	5240	14.5	15.7	6.83-22.6 / 9.46-19.9	0.0763	10107207 / EPA624	151010 / GC-MS	Acceptable
o-Xylene		WP	5250	89.5	106	65.8-134 / 77.3-122	0.920	10107207 / EPA624	151010 / GC-MS	Acceptable
Toluene	NE	WP	5140	<2.0				10107207 / EPA624	151010 / GC-MS	Acceptable
Total Xylenes	N	WP	5260	104	121	69.2-163 / 84.8-147	0.769	10107207 / EPA624	151010 / GC-MS	Acceptable

Product: Demand **WP Lot Number: 42545** **APG+ Lot Number: 42546** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range / Warn Range	ZScore	Method Code / Method Desc.	Technology Code / Technology Desc.	Evaluation
Biochemical Oxygen Demand	NE	WP	1530	41.5	51.0	25.6-76.4 / 34.1-67.9	1.12	10075408 / EPA405.1	352010 / POT	Acceptable
Carbonaceous BOD	NE	WP	1555	44.6	43.9	19.7-68.1 / 27.7-60.1	0.0866	20027401 / SM18thED5210B	352010 / POT	Acceptable
Chemical Oxygen Demand	NE	WP	1565	59.9	82.3	59.8-97.8 / 66.1-91.5	2.99	10076003 / EPA410.4	401010 / COLOR	**Check for Error**
Total Organic Carbon	NE	WP	2040	16.7	32.5	27.1-37.6 / 28.9-35.9	8.92	10078203 / EPA415.1	402010 / IR	**Not Acceptable**

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		**WP April 2005 WP #5004**
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**06/06/2005**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**04/04/2005**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**05/18/2005**

Product: Fluoride **WP Lot Number: 42474** **APG+ Lot Number: 42475** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Fluoride	NE	WP	1730	1.54	1.62	1.40-1.83	0.983	10053006	251510	Acceptable
						1.47-1.75		EPA300.0	IC	

Product: Herbicides **WP Lot Number: 42526** **APG+ Lot Number: 42527** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
2,4,5-T	N	WP	8655	0.886	3.77	0.144-5.75	2.21	10105609	152010	**Check for Error**
						1.08-4.82		EPA615	GC-ECD	
2,4,5-TP (silvex)	N	WP	8650	1.68	4.22	0.162-5.91	1.42	10105609	152010	Acceptable
						1.12-4.96		EPA615	GC-ECD	
2,4-D	N	WP	8545	0.884	4.62	0.000-7.43	2.04	10105609	152010	**Check for Error**
						0.940-6.14		EPA615	GC-ECD	
Dicamba	N	WP	8595	0.205	1.88	0.000-2.98	2.58	10105609	152010	**Check for Error**
						0.494-2.49		EPA615	GC-ECD	
Dinoseb		WP	8620	4.64	7.98	0.000-13.4	0.378	10105609	152010	Acceptable
						0.440-10.8		EPA615	GC-ECD	

Product: Hexavalent Chromium **WP Lot Number: 42496** **APG+ Lot Number: 42497** **Units: ug/L**

Analyte	Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Chromium, Hexavalent	WP	1045	358	386	314-444	0.972	10162206	401010	Acceptable
					336-422		EPA7196	COLOR	

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP April 2005 WP #5004
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	06/06/2005
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	04/04/2005
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	05/18/2005

Product: Minerals **WP Lot Number: 42566-42567** **APG+ Lot Number: 42568-42569** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Alkalinity as CaCO3	NE	WP	1505	12.1	11.4	8.23-15.9 9.46-14.5	0.0787	10054601 EPA310.1	651010 TITR	Acceptable
Calcium	NE	WP	1035	28.8	27.9	24.7-31.8 25.9-30.7	0.420	10013408 EPA200.7	102015 ICP-AES	Acceptable
Chloride	NE	WP	1575	78.8	80.2	72.5-87.7 75.0-85.2	0.512	10053006 EPA300.0	251510 IC	Acceptable
Chloride	NE	WP	1575	79.8	80.2	72.5-87.7 75.0-85.2	0.118	10057406 EPA325.3	651010 TITR	Acceptable
Conductivity	NE	WP	1610	293	331	298-346 306-338	3.56	10006209 EPA120.1	351010 COND	**Not Acceptable**
Magnesium	NE	WP	1085	2.33	2.42	2.04-2.81 2.17-2.67	0.709	10013408 EPA200.7	102015 ICP-AES	Acceptable
Potassium	NE	WP	1125	13.2	13.1	11.1-15.1 11.8-14.4	0.150	10013408 EPA200.7	102015 ICP-AES	Acceptable
Sodium	NE	WP	1155	16.4	15.9	14.1-18.2 14.8-17.6	0.288	10013408 EPA200.7	102015 ICP-AES	Acceptable
Sulfate	NE	WP	2000	5.81	6.16	3.86-8.38 4.61-7.63	0.412	10053006 EPA300.0	251510 IC	Acceptable
Sulfate	NE	WP	2000	5.2	6.16	3.86-8.38 4.61-7.63	1.22	10073606 EPA375.4	401010 COLOR	Acceptable
Total Dissolved Solids	NE	WP	1705	165	176	143-249 160-232	1.74	10009004 EPA160.1	201010 GRAV	Acceptable
Total Hardness as CaCO3	NE	WP	1755	81.5	79.7	71.1-88.9 74.1-85.9	0.505	20003401 SM1819ED2340B	102015 ICP-AES	Acceptable

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		**WP April 2005 WP #5004**
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**06/06/2005**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**04/04/2005**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**05/18/2005**

Product: Nutrients **WP Lot Number: 42615-42616** **APG+ Lot Number: 42618-42635** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Ammonia Nitrogen as N	NE	WP	1515	5.82	5.25	4.02-6.44 4.42-6.04	1.46	10063806 EPA350.2	401010 COLOR	Acceptable
Ammonia Nitrogen as N	NE	WP	1515	5.96	5.25	4.02-6.44 4.42-6.04	1.81	10064207 EPA350.3	352010 POT	Acceptable
Nitrate Nitrogen as N	NE	WP	1810	3.91	4.15	3.25-4.96 3.54-4.68	0.702	10053006 EPA300.0	251510 IC	Acceptable
Nitrate Nitrogen as N	NE	WP	1810	3.41	4.15	3.25-4.96 3.54-4.68	2.46	10067808 EPA353.3	401010 COLOR	**Check for Error**
Orthophosphate as P	NE	WP	1870	2.60	2.72	2.32-3.15 2.45-3.01	0.935	10053006 EPA300.0	251510 IC	Acceptable
Orthophosphate as P	NE	WP	1870	2.82	2.72	2.32-3.15 2.45-3.01	0.647	10070607 EPA365.3	401010 COLOR	Acceptable
Total Kjeldahl Nitrogen	NE	WP	1795		7.48	5.24-9.57 5.97-8.85				Not Reported
Total Phosphorus as P	NE	WP	1910	2.83	3.15	2.39-3.71 2.61-3.49	1.00	10070607 EPA365.3	401010 COLOR	Acceptable

Product: Oil & Grease **WP Lot Number: 42613** **APG+ Lot Number: 42614** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Oil & Grease	NE	WP	1860	17.9	20.1	11.6-24.9 13.9-22.7	0.180	10127409 EPA1664	201010 GRAV	Acceptable

Performance Summary

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APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP April 2005 WP #5004
Study Print Date 06/06/2005
Study Shipment Date 04/04/2005
Study Closing Date 05/18/2005

Product: PAH-HPLC **WP Lot Number: 42580** **APG+ Lot Number: 42581** **Units: ug/L**

Analyte	Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Acenaphthene	WP	5500	4.39	6.40	1.81-7.56 2.76-6.60	0.302	10107401 EPA625	151010 GC-MS-SIM	Acceptable
Acenaphthylene	WP	5505	<0.2				10107401 EPA625	151010 GC-MS-SIM	Acceptable
Anthracene	WP	5555	1.14	1.49	0.157-2.06 0.474-1.75	0.0943	10107401 EPA625	151010 GC-MS-SIM	Acceptable
Benzo(a)anthracene	WP	5575	1.57	1.70	0.473-2.22 0.768-1.93	0.756	10107401 EPA625	151010 GC-MS-SIM	Acceptable
Benzo(a)pyrene	WP	5580	<0.2				10107401 EPA625	151010 GC-MS-SIM	Acceptable
Benzo(b)fluoranthene	WP	5585	<0.2				10107401 EPA625	151010 GC-MS-SIM	Acceptable
Benzo(g,h,i)perylene	WP	5590	<0.2				10107401 EPA625	151010 GC-MS-SIM	Acceptable
Benzo(k)fluoranthene	WP	5600	<0.2				10107401 EPA625	151010 GC-MS-SIM	Acceptable
Chrysene	WP	5855	<0.2				10107401 EPA625	151010 GC-MS-SIM	Acceptable
Dibenzo(a,h)anthracene	WP	5895	<0.2				10107401 EPA625	151010 GC-MS-SIM	Acceptable
Fluoranthene	WP	6265	<0.2				10107401 EPA625	151010 GC-MS-SIM	Acceptable
Fluorene	WP	6270	15.2	20.4	7.00-24.1 9.82-21.2	0.106	10107401 EPA625	151010 GC-MS-SIM	Acceptable
Indeno (1,2,3-cd) pyrene	WP	6315	<0.2				10107401 EPA625	151010 GC-MS-SIM	Acceptable

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		**WP April 2005 WP #5004**
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**06/06/2005**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**04/04/2005**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**05/18/2005**

Product: PAH-HPLC **WP Lot Number: 42580** **APG+ Lot Number: 42581** **Units: ug/L**

Analyte	Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Naphthalene	WP	5005	13.1	21.1	5.42-24.2 8.54-21.1	0.543	10107401 EPA625	151010 GC-MS-SIM	Acceptable
Phenanthrene	WP	6615	1.35	1.72	0.369-2.25 0.682-1.94	0.127	10107401 EPA625	151010 GC-MS-SIM	Acceptable
Pyrene	WP	6665	1.61	1.76	0.866-2.01 1.06-1.82	0.890	10107401 EPA625	151010 GC-MS-SIM	Acceptable

Product: PCBs in Oil **WP Lot Number: 42501-42502** **Units: mg/kg**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aroclor 1016/1242 in Oil Sample 1	NE	WP	8880	40.2	36.8	6.50-49.9 13.7-42.7	1.66	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1016/1242 in Oil Sample 2	NE	WP	8880	<2				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1254 in Oil Sample 1	NE	WP	8905	<2				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1254 in Oil Sample 2	NE	WP	8905	38.4	39.6	6.25-57.7 14.8-49.2	0.746	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1260 in Oil Sample 1	NE	WP	8910	<2				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1260 in Oil Sample 2	NE	WP	8910	<2				10103603 EPA608	152010 GC-ECD	Acceptable

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		**WP April 2005 WP #5004**
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**06/06/2005**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**04/04/2005**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**05/18/2005**

Product: PCBs in Water **WP Lot Number: 42481-42482** **APG+ Lot Number: 42483** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aroclor 1016/1242 Sample 1	NE	WP	8880	<0.2				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1016/1242 Sample 2	NE	WP	8880	9.55	12.8	3.27-18.3 5.80-15.8	0.500	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1221 Sample 2		WP	8885	<0.2				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1232 Sample 1	NE	WP	8890	<0.2				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1232 Sample 2	NE	WP	8890	<0.2				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1248 Sample 1	NE	WP	8900	3.17	3.26	1.34-4.55 1.88-4.02	0.411	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1248 Sample 2	NE	WP	8900	<0.2				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1254 Sample 1	NE	WP	8905	<0.2				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1254 Sample 2	NE	WP	8905	<0.2				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1260 Sample 1	NE	WP	8910	<0.2				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1260 Sample 2	NE	WP	8910	<0.2				10103603 EPA608	152010 GC-ECD	Acceptable

Performance Summary

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APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

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913-829-0101 x26

WP April 2005 WP #5004
Study Print Date 06/06/2005
Study Shipment Date 04/04/2005
Study Closing Date 05/18/2005

Product: Pesticides (NELAC) **WP Lot Number: 42490** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
alpha-BHC	N	WP	7110	4.96	6.83	2.51-9.46 3.67-8.31	0.888	10103603 EPA608	152010 GC-ECD	Acceptable
beta-BHC	N	WP	7115	18.2	23.8	9.15-33.9 13.3-29.7	0.801	10103603 EPA608	152010 GC-ECD	Acceptable
delta-BHC	N	WP	7105	16.0	20.4	4.92-28.7 8.88-24.7	0.202	10103603 EPA608	152010 GC-ECD	Acceptable
Endosulfan I	N	WP	7510	11.8	13.6	5.62-19.8 7.98-17.4	0.381	10103603 EPA608	152010 GC-ECD	Acceptable
Endosulfan II	N	WP	7515	27.8	29.8	5.74-47.9 12.7-40.9	0.142	10103603 EPA608	152010 GC-ECD	Acceptable
Endosulfan sulfate	N	WP	7520	19.5	22.4	3.59-36.9 9.18-31.4	0.144	10103603 EPA608	152010 GC-ECD	Acceptable
Endrin	N	WP	7540	24.6	29.8	10.2-45.4 16.1-39.5	0.546	10103603 EPA608	152010 GC-ECD	Acceptable
Endrin aldehyde	N	WP	7530	11.6	10.9	3.57-16.0 5.62-13.9	0.889	10103603 EPA608	152010 GC-ECD	Acceptable
gamma-BHC	N	WP	7120	9.56	12.2	3.92-17.7 6.22-15.4	0.541	10103603 EPA608	152010 GC-ECD	Acceptable
Methoxychlor	N	WP	7810	1.43	1.40	0.191-2.93 0.648-2.47	0.285	10103603 EPA608	152010 GC-ECD	Acceptable

Performance Summary

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APG Customer 3655
EPA Lab Code KS00902
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WP April 2005 WP #5004
Study Print Date 06/06/2005
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Product: Pesticides (WP) **WP Lot Number: 42489** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
4,4'-DDD	NE	WP	7355	0.420	0.597	0.279-0.846 0.373-0.751	1.50	10103603 EPA608	152010 GC-ECD	Acceptable
4,4'-DDE	NE	WP	7360	0.423	0.628	0.288-0.855 0.383-0.761	1.58	10103603 EPA608	152010 GC-ECD	Acceptable
4,4'-DDT	NE	WP	7365	0.175	0.221	0.0816-0.330 0.123-0.289	0.749	10103603 EPA608	152010 GC-ECD	Acceptable
Aldrin	NE	WP	7025	0.420	0.727	0.183-0.985 0.316-0.852	1.22	10103603 EPA608	152010 GC-ECD	Acceptable
Dieldrin	NE	WP	7470	0.937	1.16	0.596-1.60 0.764-1.44	0.970	10103603 EPA608	152010 GC-ECD	Acceptable
Heptachlor	NE	WP	7685	1.75	2.74	0.722-3.82 1.24-3.30	1.01	10103603 EPA608	152010 GC-ECD	Acceptable
Heptachlor epoxide (beta)	NE	WP	7690	0.843	1.08	0.548-1.39 0.687-1.25	0.894	10103603 EPA608	152010 GC-ECD	Acceptable

Product: pH **WP Lot Number: 42807** **APG+ Lot Number: 42808** **Units: Units**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
pH	NE	WP	1900	9.21	9.24	8.96-9.52 9.05-9.43	0.321	10008205 EPA150.1	352010 POT	Acceptable

Performance Summary

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EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
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Nelac Point Of Contact
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WP April 2005 WP #5004
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Study Closing Date 05/18/2005

Product: Solids **WP Lot Number: 97713** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Dissolved Solids		WP	1705	54.0	69.6	43.2-98.5	1.83	10009004	201010	Acceptable
						52.4-89.3		EPA160.1	GRAV	
Total Solids		WP	1950	75.0	109	81.9-137	3.70	10009800	201010	**Not Acceptable**
						91.1-128		EPA160.3	GRAV	
Total Suspended Solids	NE	WP	1960	37.0	39.4	29.2-42.1	0.651	10009402	201010	Acceptable
						31.3-39.9		EPA160.2	GRAV	

Product: Total Chlordane **WP Lot Number: 42522** **APG+ Lot Number: 42523** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Chlordane	NE	WP	7250	9.90	12.8	5.59-18.2	0.948	10103603	152010	Acceptable
						7.68-16.1		EPA608	GC-ECD	

Product: Total Cyanide **WP Lot Number: 42564** **APG+ Lot Number: 42565** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Cyanide	NE	WP	1645	0.988	0.891	0.620-1.14	1.21	10060205	401010	Acceptable
						0.707-1.06		EPA335.2	COLOR	

Product: Total Phenolics **WP Lot Number: 42925** **APG+ Lot Number: 42908** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Phenolics	NE	WP	1905	0.684	1.30	0.471-1.25	1.35	10079604	401010	Acceptable
						0.600-1.12		EPA420.2	COLOR	

Performance Summary

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APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP April 2005 WP #5004
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	06/06/2005
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	04/04/2005
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	05/18/2005

Product: Total Residual Chlorine **WP Lot Number: 42601** **APG+ Lot Number: 42602** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Residual Chlorine	NE	WP	1940	0.850	0.940	0.672-1.09	0.445	10059004	651010	Acceptable
						0.742-1.02		EPA330.4	TITR	

Product: Toxaphene **WP Lot Number: 42524** **APG+ Lot Number: 42525** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Toxaphene	N	WP	8250	2.53	3.89	0.822-6.73	1.26	10103603	152010	Acceptable
						1.80-5.74		EPA608	GC-ECD	

Product: TPH Diesel (DRO) **WP Lot Number: 42431** **APG+ Lot Number: 42434** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
TPH Diesel (DRO)		WP	9369	0.45	1.07	0.00609-1.36	1.03	10173203	152510	Acceptable
						0.231-1.14		EPA8015	GC-FID	

Product: TPH Gasoline (GRO) **WP Lot Number: 42432** **APG+ Lot Number: 42433** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
TPH Gasoline (GRO)		WP	9408	1.04	1.05	0.356-1.52	0.526	10173203	152510	Acceptable
						0.550-1.33		EPA8015	GC-FID	
TPH Gasoline (GRO)		WP	9408	0.86	1.05	0.356-1.52	0.402	10107207	151010	Acceptable
						0.550-1.33		EPA 624	GC-MS	

Performance Summary

APG Customer 3655
EPA Lab Code KS00902
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APG NIST/NVLAP 200384-0

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WP April 2005 WP #5004
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Study Closing Date 05/18/2005

Product: Trace Metals **WP Lot Number: 42625-42626** **APG+ Lot Number: 42627-42909** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aluminum	NE	WP	1000	1690	1670	1430-1900 1510-1810	0.388	10013408 EPA200.7	102015 ICP-AES	Acceptable
Aluminum	NE	WP	1000	1700	1670	1430-1900 1510-1810	0.517	10014401 EPA200.8	102010 ICP-MS	Acceptable
Antimony	NE	WP	1005	605	584	410-703 459-653	1.01	10013408 EPA200.7	102015 ICP-AES	Acceptable
Antimony	NE	WP	1005	598	584	410-703 459-653	0.862	10014401 EPA200.8	102010 ICP-MS	Acceptable
Arsenic	NE	WP	1010	340	335	279-394 298-374	0.209	10013408 EPA200.7	102015 ICP-AES	Acceptable
Arsenic	NE	WP	1010	372	335	279-394 298-374	1.88	10014401 EPA200.8	102010 ICP-MS	Acceptable
Barium	N	WP	1015	144	140	126-164 132-158	0.159	10013408 EPA200.7	102015 ICP-AES	Acceptable
Barium	N	WP	1015	137	140	126-164 132-158	1.28	10014401 EPA200.8	102010 ICP-MS	Acceptable
Beryllium	NE	WP	1020	9.80	9.97	7.05-11.5 7.79-10.7	0.733	10013408 EPA200.7	102015 ICP-AES	Acceptable
Beryllium	NE	WP	1020	9.27	9.97	7.05-11.5 7.79-10.7	0.0136	10014401 EPA200.8	102010 ICP-MS	Acceptable
Boron		WP	1025	665	705	585-805 622-768	0.817	10013408 EPA200.7	102015 ICP-AES	Acceptable
Boron		WP	1025	639	705	585-805 622-768	1.53	10014401 EPA200.8	102010 ICP-MS	Acceptable
Cadmium	NE	WP	1030	330	327	279-372 294-356	0.323	10013408 EPA200.7	102015 ICP-AES	Acceptable

Performance Summary

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APG Customer	3655	Laboratory Address	Nelac Point Of Contact		**WP April 2005 WP #5004**
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**06/06/2005**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**04/04/2005**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**05/18/2005**

Product: Trace Metals **WP Lot Number: 42625-42626** **APG+ Lot Number: 42627-42909** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Cadmium	NE	WP	1030	322	327	279-372 294-356	0.194	10014401 EPA200.8	102010 ICP-MS	Acceptable
Chromium	NE	WP	1040	136	138	118-158 125-151	0.303	10013408 EPA200.7	102015 ICP-AES	Acceptable
Chromium	NE	WP	1040	125	138	118-158 125-151	1.97	10014401 EPA200.8	102010 ICP-MS	Acceptable
Cobalt	NE	WP	1050	399	403	354-452 370-436	0.244	10014401 EPA200.8	102010 ICP-MS	Acceptable
Cobalt	NE	WP	1050	439	403	354-452 370-436	2.20	10013408 EPA200.7	102015 ICP-AES	**Check for Error**
Copper	NE	WP	1055	848	854	777-936 804-910	0.340	10013408 EPA200.7	102015 ICP-AES	Acceptable
Copper	NE	WP	1055	838	854	777-936 804-910	0.717	10014401 EPA200.8	102010 ICP-MS	Acceptable
Iron	NE	WP	1070	1665	1630	1440-1840 1510-1770	0.381	10013408 EPA200.7	102015 ICP-AES	Acceptable
Iron	NE	WP	1070	1580	1630	1440-1840 1510-1770	0.915	10014401 EPA200.8	102010 ICP-MS	Acceptable
Lead	NE	WP	1075	110	104	84.7-123 91.1-117	0.930	10013408 EPA200.7	102015 ICP-AES	Acceptable
Lead	NE	WP	1075	104	104	84.7-123 91.1-117	0.000	10014401 EPA200.8	102010 ICP-MS	Acceptable
Manganese	NE	WP	1090	3650	3450	3100-3830 3230-3710	1.49	10013408 EPA200.7	102015 ICP-AES	Acceptable
Manganese	NE	WP	1090	3610	3450	3100-3830 3230-3710	1.16	10014401 EPA200.8	102010 ICP-MS	Acceptable

Performance Summary

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APG Customer	3655	Laboratory Address	Nelac Point Of Contact		**WP April 2005 WP #5004**
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**06/06/2005**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**04/04/2005**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**05/18/2005**

Product: Trace Metals **WP Lot Number: 42625-42626** **APG+ Lot Number: 42627-42909** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Mercury	NE	WP	1095	3.25	3.82	2.77-4.87 3.12-4.52	1.63	10036201 EPA245.1	101010 CVAAS	Acceptable
Mercury	NE	WP	1095	3.11	3.82	2.77-4.87 3.12-4.52	2.03	10014401 EPA200.8	102010 ICP-MS	**Check for Error**
Molybdenum	NE	WP	1100	486	489	420-559 444-536	0.172	10013408 EPA200.7	102015 ICP-AES	Acceptable
Molybdenum	NE	WP	1100	518	489	420-559 444-536	1.21	10014401 EPA200.8	102010 ICP-MS	Acceptable
Nickel	NE	WP	1105	1398	1480	1340-1650 1400-1600	1.99	10013408 EPA200.7	102015 ICP-AES	Acceptable
Nickel	NE	WP	1105	1430	1480	1340-1650 1400-1600	1.36	10014401 EPA200.8	102010 ICP-MS	Acceptable
Selenium	NE	WP	1140	1820	1730	1380-2000 1480-1900	1.25	10013408 EPA200.7	102015 ICP-AES	Acceptable
Selenium	NE	WP	1140	1770	1730	1380-2000 1480-1900	0.769	10014401 EPA200.8	102010 ICP-MS	Acceptable
Silver	NE	WP	1150	548	528	453-605 478-580	0.751	10013408 EPA200.7	102015 ICP-AES	Acceptable
Silver	NE	WP	1150	489	528	453-605 478-580	1.58	10014401 EPA200.8	102010 ICP-MS	Acceptable
Strontium	NE	WP	1160	280	273	232-313 246-300	0.519	10013408 EPA200.7	102015 ICP-AES	Acceptable
Strontium	NE	WP	1160	268	273	232-313 246-300	0.370	10014401 EPA200.8	102010 ICP-MS	Acceptable
Thallium	NE	WP	1165	74.9	76.0	58.6-91.3 64.1-85.9	0.0183	10013408 EPA200.7	102015 ICP-AES	Acceptable

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		**WP April 2005 WP #5004**
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**06/06/2005**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**04/04/2005**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**05/18/2005**

Product: Trace Metals **WP Lot Number: 42625-42626** **APG+ Lot Number: 42627-42909** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Thallium	NE	WP	1165	72.0	76.0	58.6-91.3 64.1-85.9	0.550	10014401 EPA200.8	102010 ICP-MS	Acceptable
Tin	N	WP	1175	1330	1350	1070-1630 1160-1540	0.212	10014401 EPA200.8	102010 ICP-MS	Acceptable
Tin	N	WP	1175	1320	1350	1070-1630 1160-1540	0.318	10013408 EPA200.7	102015 ICP-AES	Acceptable
Titanium	NE	WP	1180	272	283	244-318 256-306	0.720	10014401 EPA200.8	102010 ICP-MS	Acceptable
Titanium	NE	WP	1180	274	283	244-318 256-306	0.560	10013408 EPA200.7	102015 ICP-AES	Acceptable
Vanadium	NE	WP	1185	492	500	449-548 466-532	0.422	10014401 EPA200.8	102010 ICP-MS	Acceptable
Vanadium	NE	WP	1185	513	500	449-548 466-532	0.843	10013408 EPA200.7	102015 ICP-AES	Acceptable
Zinc	NE	WP	1190	1470	1440	1280-1620 1340-1560	0.357	10014401 EPA200.8	102010 ICP-MS	Acceptable
Zinc	NE	WP	1190	1570	1440	1280-1620 1340-1560	2.14	10013408 EPA200.7	102015 ICP-AES	**Check for Error**

Performance Summary

APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP April 2005 WP #5004
Study Print Date 06/06/2005
Study Shipment Date 04/04/2005
Study Closing Date 05/18/2005

Product: Volatiles **WP Lot Number: 42846** **APG+ Lot Number: 42847** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
1,1,1-Trichloroethane	NE	WP	5160	62.2	62.8	40.5-82.9 47.6-75.8	0.0707	10107207 EPA624	151010 GC-MS	Acceptable
1,1,2,2-Tetrachloroethane	N	WP	5110	25.4	30.1	17.2-42.2 21.3-38.1	1.03	10107207 EPA624	151010 GC-MS	Acceptable
1,1,2-Trichloroethane	N	WP	5165	117	131	88.6-171 103-157	0.949	10107207 EPA624	151010 GC-MS	Acceptable
1,1-Dichloroethane		WP	4630	7.1	10.6	6.62-15.1 8.06-13.7	2.68	10107207 EPA624	151010 GC-MS	**Check for Error**
1,1-Dichloroethene	N	WP	4640	25.3	25.0	12.4-40.2 17.0-35.6	0.216	10107207 EPA624	151010 GC-MS	Acceptable
1,2-Dichloroethane	NE	WP	4635	43.7	43.7	30.2-58.3 34.9-53.7	0.128	10107207 EPA624	151010 GC-MS	Acceptable
1,2-Dichloropropane	N	WP	4655	94.3	115	77.3-146 89.0-135	1.54	10107207 EPA624	151010 GC-MS	Acceptable
Bromodichloromethane	NE	WP	4395	8.53	8.90	6.04-11.8 7.00-10.9	0.415	10107207 EPA624	151010 GC-MS	Acceptable
Bromoform	NE	WP	4400	14.8	18.9	11.5-26.1 14.0-23.6	1.65	10107207 EPA624	151010 GC-MS	Acceptable
Bromomethane	N	WP	4950	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
Carbon tetrachloride	NE	WP	4455	33.8	32.2	20.0-45.5 24.3-41.3	0.236	10107207 EPA624	151010 GC-MS	Acceptable
Chlorobenzene	NE	WP	4475	48.8	51.9	36.6-66.1 41.6-61.2	0.528	10107207 EPA624	151010 GC-MS	Acceptable
Chloroethane	N	WP	4485	<2.0				10107207 EPA624	151010 GC-MS	Acceptable

Performance Summary

3655

APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP April 2005 WP #5004
Study Print Date 06/06/2005
Study Shipment Date 04/04/2005
Study Closing Date 05/18/2005

Product: Volatiles **WP Lot Number: 42846** **APG+ Lot Number: 42847** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Chloroform	NE	WP	4505	21.5	21.7	15.3-28.1 17.5-25.9	0.0943	10107207 EPA624	151010 GC-MS	Acceptable
Chloromethane	N	WP	4960	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
Dibromochloromethane	NE	WP	4575	58.4	63.2	41.8-84.0 48.8-77.0	0.640	10107207 EPA624	151010 GC-MS	Acceptable
Methylene chloride	NE	WP	4975	14.8	12.5	7.94-18.0 9.56-16.2	1.14	10107207 EPA624	151010 GC-MS	Acceptable
Tetrachloroethene	NE	WP	5115	36.8	38.3	25.3-49.2 29.3-45.3	0.126	10107207 EPA624	151010 GC-MS	Acceptable
trans-1,2-Dichloroethene	N	WP	4700	40.1	43.6	26.4-60.8 32.1-55.1	0.610	10107207 EPA624	151010 GC-MS	Acceptable
trans-1,3-Dichloropropene	N	WP	4685	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
Trichloroethene	NE	WP	5170	11.3	10.5	6.97-13.8 8.12-12.7	0.789	10107207 EPA624	151010 GC-MS	Acceptable
Trichlorofluoromethane	N	WP	5175	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
Vinyl Chloride	N	WP	5235	<2.0				10107207 EPA624	151010 GC-MS	Acceptable

Analytical Products Group, Inc.

PERFORMANCE REPORT

WP Performance Summary

May 2005 WP

APG Customer Code: 3655

Analytical Management Lab
Suite B
15130 South Keeler
Olathe, KS 66062

Laboratory of
EXCELLENCE

NVLAP®
LAB CODE 200384-0

Study Certification
May 2005 WP

I have reviewed the statistical analysis 285 which was performed on May 2005 WP. The statistical analysis yielded results which are consistent with NIST and NELAC Requirements.

Thomas V. Coyner
Technical Director

I have reviewed these results and find them to meet the appropriate NELAC and NIST Requirements.

Quality Assurance Manager

This report was produced at
Analytical Products Group, Inc
2730 Washington Blvd.
Belpre, Ohio 45714

This report has been produced in accordance with Analytical Products Group, Inc. ISO 9001:2000 Certification (CERT-9513-2003-AQ-HOU-RAB) issued by Det Norske Vertitas.

Water Pollution (WP) Proficiency Testing Program Report

Introduction:

The Analytical Products Group, Inc. Proficiency Testing Program has been designed to evaluate your laboratory's performance against other participating laboratories on the same set of standards. This report has been specifically prepared for your organization to assist you in ensuring that your procedures are in-control. Therefore, in most cases sample levels are not designed to challenge the detection limits of the method.

The Analytical Products Group Proficiency Testing Program is certified to meet all of the following requirements:

- For applicable analytes, this Proficiency Testing Program is accredited by NIST according to the National Voluntary Laboratory Accreditation Program (Handbook 150-19) for Chemical Calibrations: Providers of Proficiency Testing (NVLAP Code 200384-0)

- Analytical Products Group, Inc. Quality System is registered with DNVCertification to meet the ISO 9001 standard to design, manufacture, and distribute chemical reference materials and operate Proficiency Testing Programs (Certificate # 98-HOU-AQ-9513)

- For applicable analytes, this Proficiency Testing Program is certified to meet all requirements as specified in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).

Please note that this report contains data that are not covered by the NVLAP accredition. This report must not be used to claim product certification, approval, or endorsement by NVLAP, NIST, or any agency of the federal government.

Report Layout

This report contains the following sub-reports:

Introduction -This section is the general introduction to the Analytical Products Group, Inc. Proficiency Testing Program as well as all of the certifications that the organization maintains.

Terms & Definitions - Defines all of the terms used throughout the report. This is an excellent section to refer to first if you have any questions.

Laboratory Benchmark Report - This report presents an overall summary of the study as well as your performance relative to all other study participants.

Comment Report - Contains comments submitted to Analytical Products Group by the program participants or comments by Analytical Products Group to program participants concerning unusual characteristics of the data set or problems with standards which could influence the interpretation of the data. Please review this section thoroughly before examining your data.

Exception Report - This report is a summary of all of the analytes that resulted in an Evaluation of "Not Acceptable" for your laboratory. The Exception Report is designed to very quickly identify for your laboratory the areas that may require your immediate attention.

Performance Evaluation Summary Report - Provides a summary of all of your data for each analyte. This summary is formatted similar to the US EPA DMR-QA Reports.

Detail Report- This report lists all the details of your data. Also included in this section are useful graphs indicating the APG Assigned Value, the Mean, the Acceptance Limits and your Reported Value. Another graph included in this section shows your Z score for the analyte of interest.

The goal of this report is to provide your laboratory with the information you need to monitor and improve your quality. If you have any questions or feedback, please give us a call (800-272-4442). Thank you for choosing Analytical Products Group as your proficiency testing provider.

Sincerely,

Gregg Frazier
General Manager

Terms & Definitions

Average Acceptable Data - This value is the average percent of data that is within the EPA Acceptance Limits for this study. The Average Acceptable Data is reported for all laboratories as a whole as well as your laboratory as an individual. These values can be found in the Laboratory Benchmark Report.

Average Percentile for All Parameters - This is your laboratory's rank on each parameter and the related rank for that parameter. The average percentile is the average of this data across all parameters reported. It is a different measure of performance than percent acceptable data since it is affected by your relative performance on each parameter. The report provides an average for this measure for all laboratories reporting as well as your laboratory's performance.

Analyte - Name of the analyte of interest as found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) or NELAC Field of Testing List.

NELAC Analyte Code - This value is the code given to the analyte by NELAC. These codes can be found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) or the NELAC Fields of Testing List.

APG Assigned Value - This value is the calculated True Value of the standard based upon the actual composition of the standard in most cases.

Z-Score - This figure corresponds to how far your Reported Value was from the EPA Mean, reported in terms of EPA Standard Deviation units. As a point of reference, the US EPA Acceptance Limits are plus or minus three (3) EPA Standard Deviations from the EPA Mean.

APG Certified Value - This value is the result of the quality control testing performed on the sample by Analytical Products Group before the sample is shipped to the customer. Through this testing, the samples are traced against a NIST Standard Reference Material where available or an external standard if a NIST Standard Reference Material is not available. The reported uncertainty of this value is expressed as two times the standard deviation (s) divided by the square root of the number of samples (n) tested (i.e. 2s/vn). This quality control testing meets the requirements as specified by the NIST National Voluntary Laboratory Accreditation Program (NVLAP) Handbook 150-19 (Chemical Calibration: Providers of Proficiency Testing).

Acceptance Range - For applicable analytes, this range is calculated based upon the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045). For the Water Pollution Program (WP), EPA Acceptance Limits are defined as ± three (3) EPA Standard Deviations from the EPA Mean.

Mean - If the analyte is included in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then this value is the estimated mean value calculated per the Criteria Document. If the analyte is not included in the Criteria Document, then this value is the Study Mean.

Study Standard Deviation - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Standard Deviation is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Your Ranking - This is your ranking based on your Z Score for the analyte of interest among all possible rankings. It is important to note that several laboratories can achieve the same rank.

Terms & Definitions (continued)

Evaluation - This value is determined by the Acceptance Limits and the Reported Value. Evaluation may have one of following values:

Value	Description
Acceptable	The Reported Value was within the Acceptable Limits or a less than (<) value was reported AND zero (0) was included in the Acceptance Limit Range AND the numeric part of the Reported Value was less than the upper limit of the Acceptance Limit.
Not Acceptable	The Reported Value is outside of the Acceptance Limits or a less than (<) value was reported AND zero (0) is not included in the Acceptance Limits range or the numeric part of the Reported Value was greater than the upper limit of the Acceptance Limit or a greater than value (>) was reported.
Check for Error	The Reported Value is between two (2) and three (3) Standard Deviations from the Mean.
Not Evaluated	The Reported Value was a text value which could not be evaluated.
Blank	No data was reported for this analyte [the analyte was not spiked].

Inter-Laboratory Statistics - This data is a summary of all of the data from all study participants for the analyte of interest. Inter-Laboratory Statistics are only reported if a minimum number of laboratories analyzed and reported data for the analyte of interest.

Product Level - Often referred to as 'Program'. This value indicates which program the analyte of interest is a part. For Water Pollution Studies (WP), the only values that Program will have are 'WP' or 'APG+'. If the analyte and range are consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then the Program value will have a value of 'WP' and if this is not the case then the Program value will be 'APG+', indicating that the analyte and/or range was specified by Analytical Products Group.

Product Type - To the right of each analyte in the performance summary is a code that designates the scope of the APG's accreditation for that analyte. An analyte designated as "NE" is applicable to NELAC and EPA requirements and ranges. An analyte designated as "N" is applicable to NELAC requirements and ranges. An analyte designated as "E" is applicable to EPA requirements and ranges. An analyte designated as "X" or without a designation is not covered by APG's accreditations.

Rank on Percent Acceptable Data - This is your laboratories rank out of all possible ranks in the current study based on percent acceptable data. It is important to note that several laboratories can achieve the same rank.

Rank on Average Percentile Data - This value is your laboratories rank out of all possible ranks based on Your Ranking converted to a percentile. It is important to note that several laboratories can achieve the same rank.

Laboratory Pass Percentage - This is the percentage of study participants that reported values within the Acceptance Limits for the analyte of interest.

Solids Standards - Solids standards are individually prepared, therefore, each standard has a unique APG Assigned Value.

Terms & Definitions (continued)

Reported Value - This value is the result that was reported by your organization for the particular analyte. Every effort has been made to ensure correct data entry. Should you find a data entry error, please notify Analytical Products Group, Inc. and we will correct the error and re-issue the report. If the Reported Value is reported as a less than (<) figure, then the only way that the Evaluation will result in an "Acceptable" value is if the numeric part of the Reported Value (i.e. 8 if the Report Value were <8) is less than the upper limit of the Acceptance Range AND zero (0) is included in the Acceptance Range. Any other non-numeric value (i.e. BDL, ND, >10, etc.) cannot be evaluated and will return an Evaluation result of "No Evaluation". This method of evaluation is consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).

Number of Acceptable Data Points - This value is the number of data points reported by your laboratory that were within the Acceptance Limits.

Weighted Rank on Percent Acceptable Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percent Acceptable Data is the weighted rank of your laboratory based on Percent Acceptable Data and the number of analytes reported.

Weight Rank on Percentile Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percentile Data is the weighted rank of your laboratory based on Rank on Average Percentile Data and the number of analytes reported.

Study Mean - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Mean is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Laboratory Benchmark Report

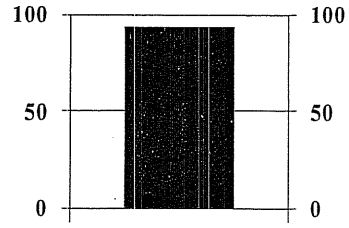
Customer Code: 3655 **May 2005 WP**

Summary of Ranking Data

Summary of National Ranking Data

Total Number of Laboratories Reporting:	87
Average Percent Acceptable Data:	93.82%
Average Percentile for all Parameters:	46.89%

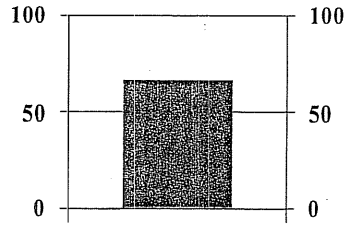
National Avg % Acceptable
93.8 %



Summary of Laboratory Ranking:

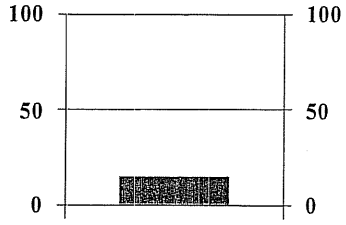
Total Data Points Reported:	3
Number of Acceptable Data Points:	2
Percent Acceptable Data:	66.67%
Average Percentile for all Parameters:	24.00%

Laboratory Avg % Acceptable
66.67%



Rank on Percent Acceptable Data:	27 of 30
Rank on Average Percentile Data:	71 of 80
Weighted Rank on Percent Acceptable Data:	38 of 41
Weighted Rank on Average Percentile Data:	70 of 83

Weighted Rank Avg Percentile
15.7 %



Exception Report

Product: Minerals **Exception Reporting** **Units: umhos/cm**

Analyte		Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Conductivity	NE	WP	1610	298	333	301-351	3.39	10006209 EPA120.1	351010 COND	Not Acceptable

Performance Summary

Performance Summary

3655

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP May 2005 WP #5005
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	06/23/2005
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	05/02/2005
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	06/15/2005

Product: Demand **WP Lot Number: 43154** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Biochemical Oxygen Demand	NE	WP	1530		30.7	15.2-46.2 20.4-41.0				Not Reported
Carbonaceous BOD	NE	WP	1555		26.5	11.9-41.1 16.7-36.3				Not Reported
Chemical Oxygen Demand	NE	WP	1565	41.0	49.4	33.2-61.8 37.9-57.1	1.36	10076003 EPA410.4	401010 COLOR	Acceptable
Total Organic Carbon	NE	WP	2040	17.9	19.5	16.2-22.8 17.3-21.7	1.45	10078203 EPA415.1	402010 IR	Acceptable

Performance Summary

3655

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP May 2005 WP #5005
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	06/23/2005
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	05/02/2005
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	06/15/2005

Product: Minerals **WP Lot Number: 43133-43134** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Alkalinity as CaCO3	NE	WP	1505		18.3	14.7-23.1 16.1-21.7				Not Reported
Calcium	NE	WP	1035		5.51	4.56-6.61 4.91-6.27				Not Reported
Chloride	NE	WP	1575		52.4	46.7-57.6 48.6-55.8				Not Reported
Conductivity	NE	WP	1610	298	333	301-351 310-342	3.39	10006209 EPA120.1	351010 COND	Not Acceptable
Magnesium	NE	WP	1085		8.96	7.78-10.1 8.16-9.72				Not Reported
Potassium	NE	WP	1125		12.0	10.2-13.9 10.8-13.2				Not Reported
Sodium	NE	WP	1155		34.5	31.0-38.4 32.2-37.2				Not Reported
Sulfate	NE	WP	2000		46.7	38.1-54.1 40.8-51.4				Not Reported
Total Dissolved Solids	NE	WP	1705		172	124-222 141-205				Not Reported
Total Hardness as CaCO3	NE	WP	1755		50.7	44.3-57.6 46.6-55.4				Not Reported

Analytical Products Group, Inc.

PERFORMANCE REPORT

WP Performance Summary

October 2005 WP

APG Customer Code: 3655

Analytical Management Lab
Suite B
15130 South Keeler
Olathe, KS 66062

Laboratory of EXCELLENCE

NVLAP

LAB CODE 200384-0

Study Certification
October 2005 WP

I have reviewed the statistical analysis 357 which was performed on October 2005 WP. The statistical analysis yielded results which are consistent with NIST and NELAC Requirements.

Thomas V. Coyner
Technical Director

I have reviewed these results and find them to meet the appropriate NELAC and NIST Requirements.

Michael D. Haller
Quality Assurance Manager

This report was produced at
Analytical Products Group, Inc
2730 Washington Blvd.
Belpre, Ohio 45714

This report has been produced in accordance with Analytical Products Group, Inc. ISO 9001:2000 Certification (CERT-9513-2003-AQ-HOU-RAB) issued by Det Norske Vertitas.

Water Pollution (WP) Proficiency Testing Program Report

Introduction:

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The goal of this report is to provide your laboratory with the information you need to monitor and improve your quality. If you have any questions or feedback, please give us a call (800-272-4442). Thank you for choosing Analytical Products Group as your proficiency testing provider.

Sincerely,

Gregg Frazier
General Manager

Terms & Definitions

Average Acceptable Data - This value is the average percent of data that is within the EPA Acceptance Limits for this study. The Average Acceptable Data is reported for all laboratories as a whole as well as your laboratory as an individual. These values can be found in the Laboratory Benchmark Report.

Average Percentile for All Parameters - This is your laboratory's rank on each parameter and the related rank for that parameter. The average percentile is the average of this data across all parameters reported. It is a different measure of performance than percent acceptable data since it is affected by your relative performance on each parameter. The report provides an average for this measure for all laboratories reporting as well as your laboratory's performance.

Analyte - Name of the analyte of interest as found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) or NELAC Field of Testing List.

NELAC Analyte Code - This value is the code given to the analyte by NELAC. These codes can be found in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) or the NELAC Fields of Testing List.

APG Assigned Value - This value is the calculated True Value of the standard based upon the actual composition of the standard in most cases.

Z-Score - This figure corresponds to how far your Reported Value was from the EPA Mean, reported in terms of EPA Standard Deviation units. As a point of reference, the US EPA Acceptance Limits are plus or minus three (3) EPA Standard Deviations from the EPA Mean.

APG Certified Value - This value is the result of the quality control testing performed on the sample by Analytical Products Group before the sample is shipped to the customer. Through this testing, the samples are traced against a NIST Standard Reference Material where available or an external standard if a NIST Standard Reference Material is not available. The reported uncertainty of this value is expressed as two times the standard deviation (s) divided by the square root of the number of samples (n) tested (i.e. 2s/vn). This quality control testing meets the requirements as specified by the NIST National Voluntary Laboratory Accreditation Program (NVLAP) Handbook 150-19 (Chemical Calibration: Providers of Proficiency Testing).

Acceptance Range - For applicable analytes, this range is calculated based upon the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045). For the Water Pollution Program (WP), EPA Acceptance Limits are defined as ± three (3) EPA Standard Deviations from the EPA Mean.

Mean - If the analyte is included in the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then this value is the estimated mean value calculated per the Criteria Document. If the analyte is not included in the Criteria Document, then this value is the Study Mean.

Study Standard Deviation - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Standard Deviation is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Your Ranking - This is your ranking based on your Z Score for the analyte of interest among all possible rankings. It is important to note that several laboratories can achieve the same rank.

Evaluation - This value is determined by the Acceptance Limits and the Reported Value. Evaluation may have one of following values:

Value	Description
Acceptable	The Reported Value was within the Acceptable Limits or a less than (<) value was reported AND zero (0) was included in the Acceptance Limit Range AND the numeric part of the Reported Value was less than the upper limit of the Acceptance Limit.
Not Acceptable	The Reported Value is outside of the Acceptance Limits or a less than (<) value was reported AND zero (0) is not included in the Acceptance Limits range or the numeric part of the Reported Value was greater than the upper limit of the Acceptance Limit or a greater than value (>) was reported.
Check for Error	The Reported Value is between two (2) and three (3) Standard Deviations from the Mean.
Not Evaluated	The Reported Value was a text value which could not be evaluated.
Blank	No data was reported for this analyte [the analyte was not spiked].

Inter-Laboratory Statistics - This data is a summary of all of the data from all study participants for the analyte of interest. Inter-Laboratory Statistics are only reported if a minimum number of laboratories analyzed and reported data for the analyte of interest.

Product Level - Often referred to as 'Program'. This value indicates which program the analyte of interest is a part. For Water Pollution Studies (WP), the only values that Program will have are 'WP' or 'APG+'. If the analyte and range are consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045) then the Program value will have a value of 'WP' and if this is not the case then the Program value will be 'APG+', indicating that the analyte and/or range was specified by Analytical Products Group.

Product Type - To the right of each analyte in the performance summary is a code that designates the scope of the APG's accreditation for that analyte. An analyte designated as "NE" is applicable to NELAC and EPA requirements and ranges. An analyte designated as "N" is applicable to NELAC requirements and ranges. An analyte designated as "E" is applicable to EPA requirements and ranges. An analyte designated as "X" or without a designation is not covered by APG's accreditations.

Rank on Percent Acceptable Data - This is your laboratories rank out of all possible ranks in the current study based on percent acceptable data. It is important to note that several laboratories can achieve the same rank.

Rank on Average Percentile Data - This value is your laboratories rank out of all possible ranks based on Your Ranking converted to a percentile. It is important to note that several laboratories can achieve the same rank.

Laboratory Pass Percentage - This is the percentage of study participants that reported values within the Acceptance Limits for the analyte of interest.

Solids Standards - Solids standards are individually prepared, therefore, each standard has a unique APG Assigned Value.

Reported Value - This value is the result that was reported by your organization for the particular analyte. Every effort has been made to ensure correct data entry. Should you find a data entry error, please notify Analytical Products Group, Inc. and we will correct the error and re-issue the report. If the Reported Value is reported as a less than (<) figure, then the only way that the Evaluation will result in an "Acceptable" value is if the numeric part of the Reported Value (i.e. 8 if the Report Value were <8) is less than the upper limit of the Acceptance Range AND zero (0) is included in the Acceptance Range. Any other non-numeric value (i.e. BDL, ND, >10, etc.) cannot be evaluated and will return an Evaluation result of "No Evaluation". This method of evaluation is consistent with the US EPA National Standards for Water Proficiency Testing Program Criteria Document (NERL-Ci-0045).

Number of Acceptable Data Points - This value is the number of data points reported by your laboratory that were within the Acceptance Limits.

Weighted Rank on Percent Acceptable Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percent Acceptable Data is the weighted rank of your laboratory based on Percent Acceptable Data and the number of analytes reported.

Weight Rank on Percentile Data - Because the Analytical Products Group Proficiency Testing Program includes laboratories of many sizes and capabilities, we have provided a weighted ranking system that will result in meaningful rankings for all study participants. The Weighted Rank on Percentile Data is the weighted rank of your laboratory based on Rank on Average Percentile Data and the number of analytes reported.

Study Mean - This value is calculated using all reported values after the removal of outliers, which are removed using standard statistical methods. The Study Mean is only reported if a statistically acceptable number of laboratories report numeric results for the analyte.

Laboratory Benchmark Report

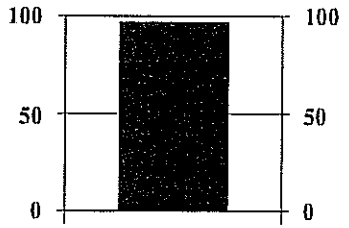
Customer Code: 3655 **October 2005 WP**

Summary of Ranking Data

Summary of National Ranking Data

Total Number of Laboratories Reporting:	320
Average Percent Acceptable Data:	96.21%
Average Percentile for all Parameters:	30.41%

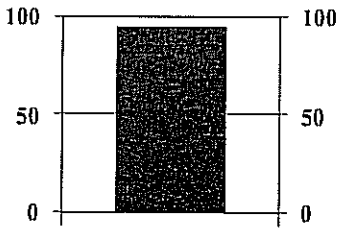
National Avg % Acceptable
96.2 %



Summary of Laboratory Ranking:

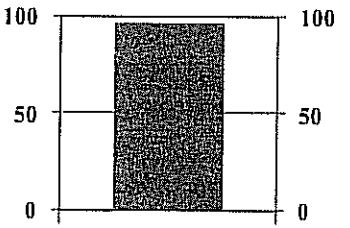
Total Data Points Reported:	306
Number of Acceptable Data Points:	290
Percent Acceptable Data:	94.77%
Average Percentile for all Parameters:	54.55%

Laboratory Avg % Acceptable
94.77%



Rank on Percent Acceptable Data:	36 of 78
Rank on Average Percentile Data:	149 of 288
Weighted Rank on Percent Acceptable Data:	8 of 79
Weighted Rank on Average Percentile Data:	9 of 293

Weighted Rank Avg Percentile
96.9 %



Comment Report

There are no comments to report.

Exception Report

Product: Diesel Range Organics (DRO) **Exception Reporting** **Units: ug/L**

Analyte		Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Diesel Range Organics (DRO)	N	WP	9369	1.41	3450	850-4450	4.42	10173203 EPA8015	152510 GC-FID	Not Acceptable

Product: Gasoline Range Organics (GRO) **Exception Reporting** **Units: ug/L**

Analyte		Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Gasoline Range Organics (GRO)	N	WP	9408	0.500	435	181-792	4.77	OK GRO GC-FID		Not Acceptable
Gasoline Range Organics (GRO)	N	WP	9408	0.577	435	181-792	4.77	10173203 EPA8015		Not Acceptable
Gasoline Range Organics (GRO)	N	WP	9408	0.476	435	181-792	4.77	624 GC/MS		Not Acceptable

Product: Herbicides **Exception Reporting** **Units: ug/L**

Analyte		Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
2,4-D	N	WP	8545	0.333	4.97	0.497-8.16	3.13	10105609 EPA615	152010 GC-ECD	Not Acceptable

Product: Nutrients **Exception Reporting** **Units: mg/L**

Analyte		Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Nitrate Nitrogen as N	NE	WP	1810	5.34	7.06	5.50-8.53	3.32	10067808 EPA353.3	401010 COLOR	Not Acceptable
Total Phosphorus as P	NE	WP	1910	6.03	4.13	3.38-4.94	7.19	10070607 EPA365.3	401010 COLOR	Not Acceptable

Product: PAH-HPLC **Exception Reporting** **Units: ug/L**

Analyte		Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation

Product: PAH-HPLC

Exception Reporting **Units: ug/L**

Analyte		Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Benzo(a)anthracene	N	WP	5575	0.529	1.27	0.781-1.44	5.30	10187607 EPA8310	251020 HPLC-UV	Not Acceptable
Benzo(b)fluoranthene	N	WP	5585	1.23	0.741	0.341-0.968	5.50	10107401 EPA625	151010 GC-MS-SIM	Not Acceptable
Benzo(b)fluoranthene	N	WP	5585	0.321	0.741	0.341-0.968	3.19	10187607 EPA8310	251020 HPLC-UV	Not Acceptable
Benzo(k)fluoranthene	N	WP	5600	0.498	1.16	0.630-1.42	4.01	10187607 EPA8310	251020 HPLC-UV	Not Acceptable
Fluoranthene	N	WP	6265	0.576	1.39	0.815-1.68	4.66	10187607 EPA8310	251020 HPLC-UV	Not Acceptable
Phenanthrene	N	WP	6615	0.273	0.641	0.318-0.836	3.52	10187607 EPA8310	251020 HPLC-UV	Not Acceptable
Pyrene	N	WP	6665	0.494	1.05	0.611-1.27	4.06	10187607 EPA8310	251020 HPLC-UV	Not Acceptable

Product: Trace Metals

Exception Reporting **Units: ug/L**

Analyte		Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Arsenic	NE	WP	1010	150	122	98.4-146	3.56	10014401 EPA200.8	102010 ICP-MS	Not Acceptable

Product: Volatiles

Exception Reporting **Units: ug/L**

Analyte		Product Level	Analyte Code	Reported Value	Assigned Value	Acceptance Range	Z-Score	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Acrolein		WP	4325	13.5				10107207 EPA624	151010 GC-MS	Not Acceptable

Performance Summary

Performance Summary

APG Customer 3655	Laboratory Address	Nelac Point Of Contact	**WP October 2005 WP #5010**
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date 12/05/2005**
Primary AA E-10254	15130 South Keeler	QA Director	**Study Shipment Date 10/03/2005**
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date 11/16/2005**

Product: Acid Extractables **Lot Number: 44616** **APG+ Lot Number: 44617** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
2,4,5-Trichlorophenol	N	WP	6835	113	138	48.2-175 69.4-154	0.0558	10107401 EPA625	151010 GC-MS	Acceptable
2,4,6-Trichlorophenol	N	WP	6840	70.4	75.1	24.0-96.2 36.0-84.1	0.859	10107401 EPA625	151010 GC-MS	Acceptable
2,4-Dichlorophenol	N	WP	6000	47.9	60.0	18.2-77.0 28.0-67.2	0.0319	10107401 EPA625	151010 GC-MS	Acceptable
2,4-Dimethylphenol	N	WP	6130	75.3	124	26.8-163 49.5-140	0.857	10107401 EPA625	151010 GC-MS	Acceptable
2,4-Dinitrophenol	N	WP	6175	113	145	14.5-198 45.1-167	0.221	10107401 EPA625	151010 GC-MS	Acceptable
2,6-Dichlorophenol	N	WP	6005	<5.0				10107401 EPA625	151010 GC-MS	Acceptable
2-Chlorophenol	N	WP	5800	110	149	42.2-187 66.3-163	0.189	10107401 EPA625	151010 GC-MS	Acceptable
2-Methyl-4,6-Dinitrophenol	N	WP	6360	101	113	37.1-159 57.4-139	0.146	10107401 EPA625	151010 GC-MS	Acceptable
2-Methylphenol	N	WP	6400	<9.5				10107401 EPA625	151010 GC-MS	Acceptable
2-Nitrophenol	N	WP	6490	109	132	31.1-173 54.7-149	0.304	10107401 EPA625	151010 GC-MS	Acceptable
3 & 4-Methylphenol	N	WP	6410	<5.0				10107401 EPA625	151010 GC-MS	Acceptable
4-Chloro-3-methylphenol	N	WP	5700	64.0	82.9	31.7-107 44.2-94.1	0.413	10107401 EPA625	151010 GC-MS	Acceptable
4-Nitrophenol	N	WP	6500	24.5	152	15.2-204 46.7-173	2.70	10107401 EPA625	151010 GC-MS	**Check for Error**

Performance Summary

APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP October 2005 WP #5010
Study Print Date 12/05/2005
Study Shipment Date 10/03/2005
Study Closing Date 11/16/2005

Product: Acid Extractables **Lot Number: 44616** **APG+ Lot Number: 44617** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Benzoic Acid		WP	5610	<5.0				10107401 EPA625	151010 GC-MS	Acceptable
Pentachlorophenol	N	WP	6605	93.0	103	26.3-142 45.6-123	0.448	10107401 EPA625	151010 GC-MS	Acceptable
Phenol	N	WP	6625	56.0	123	12.3-166 37.9-140	1.29	10107401 EPA625	151010 GC-MS	Acceptable

Product: Acidity as CaCO3 **Lot Number: 44744** **Units: mg/l**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Acidity as CaCO3	N	WP	1500	1450	1330	1190-1460 1240-1420	2.78	10054009 EPA305.1		Check for Error

Performance Summary

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APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP October 2005 WP #5010
Study Print Date 12/05/2005
Study Shipment Date 10/03/2005
Study Closing Date 11/16/2005

Product: Base Neutral Extractables **Lot Number: 44768** **APG+ Lot Number: 44769** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
1,2,4-Trichlorobenzene	N	WP	5155	<5.0				10107401 EPA625	151010 GC-MS	Acceptable
1,2-Dichlorobenzene	N	WP	4610	71.6	101	12.3-121 30.4-103	0.280	10107401 EPA625	151010 GC-MS	Acceptable
1,3-Dichlorobenzene	N	WP	4615	<4.5				10107401 EPA625	151010 GC-MS	Acceptable
1,4-Dichlorobenzene	N	WP	4620	33.0	44.2	4.42-56.5 13.1-47.8	0.293	10107401 EPA625	151010 GC-MS	Acceptable
2,4-Dinitrotoluene (2,4-DNT)	N	WP	6185	23.8	26.7	7.96-37.3 12.9-32.4	0.241	10107401 EPA625	151010 GC-MS	Acceptable
2,6-Dinitrotoluene (2,6-DNT)	N	WP	6190	131	167	70.3-209 93.3-186	0.367	10107401 EPA625	151010 GC-MS	Acceptable
2-Chloronaphthalene	N	WP	5795	21.1	25.1	7.00-31.7 11.1-27.6	0.422	10107401 EPA625	151010 GC-MS	Acceptable
2-Methylnaphthalene	N	WP	6385	<3.5				10107401 EPA625	151010 GC-MS	Acceptable
2-Nitroaniline		WP	6460	<10				10107401 EPA625	151010 GC-MS	Acceptable
3,3'-Dichlorobenzidine	N	WP	5945	<10				10107401 EPA625	151010 GC-MS	Acceptable
4-Bromophenyl phenyl ether	N	WP	5660	<8.1				10107401 EPA625	151010 GC-MS	Acceptable
4-Chloroaniline		WP	5745	<10				10107401 EPA625	151010 GC-MS	Acceptable
4-Chlorophenyl-phenylether	N	WP	5825	<9.9				10107401 EPA625	151010 GC-MS	Acceptable

Performance Summary

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APG Customer 3655	Laboratory Address	Nelac Point Of Contact	WP October 2005 WP #5010
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date 12/05/2005
Primary AA E-10254	15130 South Keeler	QA Director	Study Shipment Date 10/03/2005
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date 11/16/2005

Product: Base Neutral Extractables **Lot Number: 44768** **APG+ Lot Number: 44769** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
4-Nitroaniline		WP	6470	<10				10107401 EPA625	151010 GC-MS	Acceptable
Acenaphthene	N	WP	5500	18.8	21.2	9.90-27.4 12.8-24.5	0.0502	10107401 EPA625	151010 GC-MS	Acceptable
Acenaphthylene	N	WP	5505	124	170	68.4-205 91.1-182	0.551	10107401 EPA625	151010 GC-MS	Acceptable
Anthracene	N	WP	5555	18.7	20.2	9.13-27.2 12.1-24.2	0.170	10107401 EPA625	151010 GC-MS	Acceptable
Benzidine	N	WP	5595	<20				10107401 EPA625	151010 GC-MS	Acceptable
Benzo(a)anthracene	N	WP	5575	133	166	76.0-210 98.3-187	0.440	10107401 EPA625	151010 GC-MS	Acceptable
Benzo(a)pyrene	N	WP	5580	22.8	24.9	7.84-34.2 12.2-29.8	0.408	10107401 EPA625	151010 GC-MS	Acceptable
Benzo(b)fluoranthene	N	WP	5585	28.2	30.2	9.99-42.2 15.4-36.8	0.391	10107401 EPA625	151010 GC-MS	Acceptable
Benzo(g,h,i)perylene	N	WP	5590	<2.9				10107401 EPA625	151010 GC-MS	Acceptable
Benzo(k)fluoranthene	N	WP	5600	27.6	32.1	7.08-49.7 14.2-42.6	0.111	10107401 EPA625	151010 GC-MS	Acceptable
Benzyl alcohol		WP	5630	<10				10107401 EPA625	151010 GC-MS	Acceptable
Benzyl butyl phthalate	N	WP	5670	54.2	61.7	7.54-92.0 21.6-77.9	0.314	10107401 EPA625	151010 GC-MS	Acceptable
bis(2-Chloroethoxy) methane	N	WP	5760	<3.6				10107401 EPA625	151010 GC-MS	Acceptable

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP October 2005 WP #5010
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	12/05/2005
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/03/2005
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/16/2005

Product: Base Neutral Extractables **Lot Number:** 44768 **APG+ Lot Number:** 44769 **Units:** ug/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
bis(2-Chloroethyl) ether	N	WP	5765	<4.8				10107401 EPA625	151010 GC-MS	Acceptable
bis(2-Chloroisopropyl) ether	N	WP	5780	<9.6				10107401 EPA625	151010 GC-MS	Acceptable
bis(2-Ethylhexyl) phthalate	N	WP	6255	91.7	133	39.0-181 62.6-157	0.768	10107401 EPA625	151010 GC-MS	Acceptable
Carbazole		WP	5680	<10				10107401 EPA625	151010 GC-MS	Acceptable
Chrysene	N	WP	5855	118	166	64.4-212 89.0-187	0.819	10107401 EPA625	151010 GC-MS	Acceptable
Di-n-butylphthalate	N	WP	5925	97.3	102	33.4-133 50.0-117	0.838	10107401 EPA625	151010 GC-MS	Acceptable
Di-n-octylphthalate	N	WP	6200	47.4	57.4	16.3-86.8 28.1-75.1	0.355	10107401 EPA625	151010 GC-MS	Acceptable
Dibenz(a,h) anthracene	N	WP	5895	<4.9				10107401 EPA625	151010 GC-MS	Acceptable
Dibenzofuran	N	WP	5905	<11				10107401 EPA625	151010 GC-MS	Acceptable
Diethyl phthalate	N	WP	6070	84.6	105	19.1-145 40.1-124	0.123	10107401 EPA625	151010 GC-MS	Acceptable
Dimethyl phthalate	N	WP	6135	110	138	13.8-199 44.7-168	0.117	10107401 EPA625	151010 GC-MS	Acceptable
Fluoranthene	N	WP	6265	31.7	34.0	16.3-45.2 21.1-40.4	0.203	10107401 EPA625	151010 GC-MS	Acceptable
Fluorene	N	WP	6270	32.8	38.5	14.4-50.3 20.4-44.4	0.0716	10107401 EPA625	151010 GC-MS	Acceptable

Performance Summary

APG Customer 3655	Laboratory Address	Nelac Point Of Contact	WP October 2005 WP #5010
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Product: Base Neutral Extractables **Lot Number: 44768** **APG+ Lot Number: 44769** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Hexachlorobenzene	N	WP	6275	136	161	70.8-195 91.4-174	0.155	10107401 EPA625	151010 GC-MS	Acceptable
Hexachlorobutadiene	N	WP	4835	<5.0				10107401 EPA625	151010 GC-MS	Acceptable
Hexachlorocyclopentadiene	N	WP	6285	<10				10107401 EPA625	151010 GC-MS	Acceptable
Hexachloroethane	N	WP	4840	109	171	18.2-199 48.3-169	0.0147	10107401 EPA625	151010 GC-MS	Acceptable
Indeno (1,2,3-cd) pyrene	N	WP	6315	<4.3				10107401 EPA625	151010 GC-MS	Acceptable
Isophorone	N	WP	6320	72.3	90.6	35.7-117 49.3-104	0.302	10107401 EPA625	151010 GC-MS	Acceptable
N-nitrosodi-n-propylamine	N	WP	6545	<4.8				10107401 EPA625	151010 GC-MS	Acceptable
N-nitrosodimethylamine	N	WP	6530	<7.5				10107401 EPA625	151010 GC-MS	Acceptable
N-nitrosodiphenylamine	N	WP	6535	<6.4				10107401 EPA625	151010 GC-MS	Acceptable
Naphthalene	N	WP	5005	108	166	42.2-195 67.7-169	0.412	10107401 EPA625	151010 GC-MS	Acceptable
Nitrobenzene (NB)	N	WP	5015	47.2	58.7	18.9-73.0 27.9-64.0	0.136	10107401 EPA625	151010 GC-MS	Acceptable
Phenanthrene	N	WP	6615	29.7	32.5	16.7-42.5 21.0-38.2	0.0252	10107401 EPA625	151010 GC-MS	Acceptable
Pyrene	N	WP	6665	135	171	57.1-229 85.6-200	0.274	10107401 EPA625	151010 GC-MS	Acceptable

Performance Summary

APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
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Laboratory Address
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Nelac Point Of Contact
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Product: Base Neutral Extractables Lot Number: 44768 APG+ Lot Number: 44769 Units: ug/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Pyridine		WP	5095	<10				10107401 EPA625	151010 GC-MS	Acceptable

Product: Bromide Lot Number: 44698 APG+ Lot Number: 44699 Units: mg/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Bromide	N	WP	1540	8.83	9.40	7.99-10.8 8.46-10.3	1.21	10053006 EPA300.0		Acceptable

Product: Demand Lot Number: 44639 APG+ Lot Number: 44640 Units: mg/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Biochemical Oxygen Demand	NE	WP	1530	35.4	44.5	22.3-66.7 29.7-59.3	1.23	10075408 EPA405.1	352010 POT	Acceptable
Carbonaceous BOD	NE	WP	1555	33.6	38.4	17.2-59.6 24.3-52.5	0.679	20027401 SM18thED5210B	352010 POT	Acceptable
Chemical Oxygen Demand	NE	WP	1565	72.8	71.8	51.3-86.3 57.1-80.5	0.685	10076003 EPA410.4	401010 COLOR	Acceptable
Total Organic Carbon	NE	WP	2040	28.8	28.4	23.6-33.0 25.2-31.4	0.327	10078203 EPA415.1	402010 IR	Acceptable

Product: Diesel Range Organics (DRO) Lot Number: 44602 APG+ Lot Number: 44603 Units: ug/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Diesel Range Organics (DRO)	N	WP	9369	1.41	3450	850-4450 1450-3850	4.42	10173203 EPA8015	152510 GC-FID	Not Acceptable

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Product: DRO (Wisconsin) **Lot Number: 44759** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Diesel Range Organics (WI)	X	WP			465	230-656 290-597				Not Reported

Product: Fluoride **Lot Number: 44589** **APG+ Lot Number: 44590** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Fluoride	NE	WP	1730	1.34	1.35	1.06-1.64 1.16-1.55	0.110	10053006 EPA300.0	251510 IC	Acceptable

Product: Gasoline Range Organics (GRO) **Lot Number: 44572** **APG+ Lot Number: 44574** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Gasoline Range Organics (GRO)	N	WP	9408	0.476	435	181-792 282-690	4.77	624 GC/MS		Not Acceptable
Gasoline Range Organics (GRO)	N	WP	9408	0.500	435	181-792 282-690	4.77	OK GRO GC-FID		Not Acceptable
Gasoline Range Organics (GRO)	N	WP	9408	0.577	435	181-792 282-690	4.77	10173203 EPA8015		Not Acceptable

Product: GRO (Wisconsin) **Lot Number: 44760** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Gasoline Range Organics (WI)	X	WP			334	246-426 271-401				Not Reported

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APG Customer 3655	Laboratory Address	Nelac Point Of Contact	**WP October 2005 WP #5010**
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date 12/05/2005**
Primary AA E-10254	15130 South Keeler	QA Director	**Study Shipment Date 10/03/2005**
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date 11/16/2005**

Product: Herbicides **Lot Number: 44701** **APG+ Lot Number: 44702** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
2,4,5-T	N	WP	8655	0.867	6.13	0.613-9.05 2.02-7.64	2.82	10105609 EPA615	152010 GC-ECD	**Check for Error**
2,4,5-TP (silvex)	N	WP	8650	1.16	3.27	0.327-4.94 1.10-4.17	1.92	10105609 EPA615	152010 GC-ECD	Acceptable
2,4-D	N	WP	8545	0.333	4.97	0.497-8.16 1.77-6.88	3.13	10105609 EPA615	152010 GC-ECD	**Not Acceptable**
2,4-DB		WP	8560	<0.1				10105609 EPA615		Acceptable
3,5-Dichlorobenzoic acid		WP	8600	<0.1				10105609 EPA615		Acceptable
4-Nitrophenol		WP	6500	<0.1				10105609 EPA615		Acceptable
Acifluorfen		WP	8505	<0.1				10105609 EPA615		Acceptable
Chloramben		WP	8540	<0.1				10105609 EPA615		Acceptable
Dacthal diacid (DCPA)		WP	8550	<0.1				10105609 EPA615		Acceptable
Dalapon		WP	8555	<0.1				10105609 EPA615		Acceptable
Dicamba	N	WP	8595	1.87	7.26	0.726-10.5 2.35-8.87	2.30	10105609 EPA615	152010 GC-ECD	**Check for Error**
Dichloroprop		WP	8605	<0.1				10105609 EPA615		Acceptable
Dinoseb		WP	8620	1.09	3.56	0.000-5.84 0.326-4.74	1.31	10105609 EPA615	152010 GC-ECD	Acceptable

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APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP October 2005 WP #5010
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	12/05/2005
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/03/2005
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/16/2005

Product: Herbicides **Lot Number: 44701** **APG+ Lot Number: 44702** **Units: ug/L**

Analyte	Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Pentachlorophenol	WP	6605	<0.1				10105609 EPA615		Acceptable
Picloram	WP	8645	<0.1				10105609 EPA615		Acceptable

Product: Hexavalent Chromium **Lot Number: 44567** **APG+ Lot Number: 44568** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Chromium, Hexavalent	N	WP	1045	668	661	539-777 579-737	0.249	10162206 EPA7196	401010 COLOR	Acceptable

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APG Customer 3655	Laboratory Address	Nelac Point Of Contact	WP October 2005 WP #5010
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Product: Minerals **Lot Number: 44689-44690** **APG+ Lot Number: 44692-44693** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Alkalinity as CaCO3	NE	WP	1505	76.9	77.4	68.2-85.7 71.1-82.8	0.00867	10054601 EPA310.1	651010 TITR	Acceptable
Calcium	NE	WP	1035	9.49	9.13	7.82-10.7 8.30-10.2	0.488	10013408 EPA200.7	102015 ICP-AES	Acceptable
Calcium Hardness as CaCO3	N	WP	1550	23.7	22.8	19.5-26.7 20.7-25.5	0.489	20003401 SM1819ED2340B		Acceptable
Chloride	NE	WP	1575	86.3	87.5	75.0-100 79.2-95.9	0.303	10053006 EPA300.0	251510 IC	Acceptable
Chloride	NE	WP	1575	88.6	87.5	75.0-100 79.2-95.9	0.247	10057406 EPA325.3	651010 TITR	Acceptable
Conductivity	NE	WP	1610	600	584	525-642 545-623	0.846	10006209 EPA120.1	351010 COND	Acceptable
Magnesium	NE	WP	1085	7.00	6.98	5.91-7.98 6.25-7.64	0.160	10013408 EPA200.7	102015 ICP-AES	Acceptable
Potassium	NE	WP	1125	28.1	28.1	23.2-33.5 24.9-31.8	0.137	10013408 EPA200.7	102015 ICP-AES	Acceptable
Sodium	NE	WP	1155	79.7	79.6	67.6-91.2 71.5-87.3	0.0744	10013408 EPA200.7	102015 ICP-AES	Acceptable
Sulfate	NE	WP	2000	52.3	57.5	47.2-66.2 50.4-63.1	1.39	10053006 EPA300.0	251510 IC	Acceptable
Sulfate	NE	WP	2000	53.0	57.5	47.2-66.2 50.4-63.1	1.17	10073606 EPA375.4	401010 COLOR	Acceptable
Total Dissolved Solids	NE	WP	1705	307	316	230-383 256-357	0.0140	10009004 EPA160.1	201010 GRAV	Acceptable
Total Hardness as CaCO3	NE	WP	1755	52.6	51.6	43.9-59.6 46.5-57.0	0.339	20003401 SM1819ED2340B	102015 ICP-AES	Acceptable

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APG Customer 3655	Laboratory Address	Nelac Point Of Contact	**WP October 2005 WP #5010**
EPA Lab Code KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date 12/05/2005**
Primary AA E-10254	15130 South Keeler	QA Director	**Study Shipment Date 10/03/2005**
APG NIST/NVLAP 200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date 11/16/2005**

Product: Nitrate-Nitrite as N **Lot Number: 44745** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Nitrate-Nitrite as N	N	WP	1820	25.0	27.1	22.1-31.5 23.7-29.9	1.14	10067808 EPA353.3		Acceptable
Nitrate-Nitrite as N	N	WP	1820	25.0	27.1	22.1-31.5 23.7-29.9	1.14	10053006 EPA300.0		Acceptable

Product: Nitrite as N **Lot Number: 44675** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Nitrite as N	N	WP	1840	3.05	3.40	2.90-3.91 3.06-3.74	2.09	10068403 EPA354.1		**Check for Error**
Nitrite as N	N	WP	1840	3.44	3.40	2.90-3.91 3.06-3.74	0.229	10053006 EPA300.0		Acceptable

Performance Summary

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Product: Nutrients **Lot Number: 44794-44795** **APG+ Lot Number: 44796-44797** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Ammonia Nitrogen as N	NE	WP	1515	11.0	9.43	6.97-11.8 7.77-11.0	2.01	10064207 EPA350.3	352010 POT	Acceptable
Ammonia Nitrogen as N	NE	WP	1515	11.1	9.43	6.97-11.8 7.77-11.0	2.13	10063806 EPA350.2	401010 COLOR	**Check for Error**
Nitrate Nitrogen as N	NE	WP	1810	5.34	7.06	5.50-8.53 6.00-8.02	3.32	10067808 EPA353.3	401010 COLOR	**Not Acceptable**
Nitrate Nitrogen as N	NE	WP	1810	6.36	7.06	5.50-8.53 6.00-8.02	1.30	10053006 EPA300.0	251510 IC	Acceptable
Orthophosphate as P	NE	WP	1870	4.89	4.77	3.94-5.64 4.22-5.35	0.361	10070607 EPA365.3	401010 COLOR	Acceptable
Orthophosphate as P	NE	WP	1870	4.81	4.77	3.94-5.64 4.22-5.35	0.0781	10053006 EPA300.0	251510 IC	Acceptable
Total Kjeldahl Nitrogen	NE	WP	1795		14.3	9.47-18.5 11.0-17.0				Not Reported
Total Phosphorus as P	NE	WP	1910	6.03	4.13	3.38-4.94 3.64-4.68	7.19	10070607 EPA365.3	401010 COLOR	**Not Acceptable**

Product: Oil & Grease **Lot Number: 44709** **APG+ Lot Number: 44710** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Oil & Grease	NE	WP	1860	51.5	49.7	31.9-60.7 36.7-55.9	1.08	10127409 EPA1664	201010 GRAV	Acceptable

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APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP October 2005 WP #5010
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	12/05/2005
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/03/2005
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/16/2005

Product: PAH-GC & GCMS **Lot Number: 44636** **APG+ Lot Number: 44635** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Acenaphthene	N	WP	5500		65.3	26.5-78.6 35.2-69.9				Not Reported
Acenaphthylene	N	WP	5500		19.1	6.68-25.2 9.77-22.1				Not Reported
Anthracene	N	WP	5555		50.4	21.6-64.1 28.7-57.0				Not Reported
Benzo(a)anthracene	N	WP	5575		40.6	18.1-52.0 23.7-46.4				Not Reported
Benzo(a)pyrene	N	WP	5580		29.2	9.09-39.4 14.1-34.4				Not Reported
Benzo(b)fluoranthene	N	WP	5585		37.5	13.0-51.8 19.4-45.3				Not Reported
Benzo(g,h,i)perylene	N	WP	5590		36.2	10.2-52.1 17.2-45.1				Not Reported
Benzo(k)fluoranthene	N	WP	5600		51.7	12.3-76.8 23.0-66.0				Not Reported
Fluoranthene	N	WP	6265		72.9	32.8-89.6 42.2-80.1				Not Reported
Fluorene	N	WP	6270		53.9	21.6-67.6 29.3-59.9				Not Reported
Naphthalene	N	WP	5005		46.9	14.4-58.7 21.8-51.3				Not Reported
Phenanthrene	N	WP	6615		45.2	22.2-57.2 28.0-51.4				Not Reported
Pyrene	N	WP	6665		49.0	16.0-69.7 24.9-60.7				Not Reported

Performance Summary

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Product: PAH-HPLC **Lot Number: 44633** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Acenaphthene	N	WP	5500	1.13	2.62	0.678-3.65 1.17-3.16	2.09	10187607 EPA8310	251020 HPLC-UV	**Check for Error**
Acenaphthene	N	WP	5500	1.68	2.62	0.678-3.65 1.17-3.16	0.977	10107401 EPA625	151010 GC-MS-SIM	Acceptable
Acenaphthylene	N	WP	5505	1.36	2.93	1.28-3.61 1.67-3.22	2.79	10187607 EPA8310	251020 HPLC-UV	**Check for Error**
Acenaphthylene	N	WP	5505	1.95	2.93	1.28-3.61 1.67-3.22	1.27	10107401 EPA625	151010 GC-MS-SIM	Acceptable
Anthracene	N	WP	5555	0.372	0.906	0.258-1.26 0.424-1.09	2.31	10187607 EPA8310	251020 HPLC-UV	**Check for Error**
Anthracene	N	WP	5555	0.56	0.906	0.258-1.26 0.424-1.09	1.19	10107401 EPA625	151010 GC-MS-SIM	Acceptable
Benzo(a)anthracene	N	WP	5575	0.529	1.27	0.781-1.44 0.891-1.33	5.30	10187607 EPA8310	251020 HPLC-UV	**Not Acceptable**
Benzo(a)anthracene	N	WP	5575	1.07	1.27	0.781-1.44 0.891-1.33	0.362	10107401 EPA625	151010 GC-MS-SIM	Acceptable
Benzo(a)pyrene	N	WP	5580	0.76	1.31	0.539-1.65 0.724-1.47	1.81	10107401 EPA625	151010 GC-MS-SIM	Acceptable
Benzo(a)pyrene	N	WP	5580	0.621	1.31	0.539-1.65 0.724-1.47	2.56	10187607 EPA8310	251020 HPLC-UV	**Check for Error**
Benzo(b)fluoranthene	N	WP	5585	1.23	0.741	0.341-0.968 0.445-0.864	5.50	10107401 EPA625	151010 GC-MS-SIM	**Not Acceptable**
Benzo(b)fluoranthene	N	WP	5585	0.321	0.741	0.341-0.968 0.445-0.864	3.19	10187607 EPA8310	251020 HPLC-UV	**Not Acceptable**
Benzo(g,h,i)perylene	N	WP	5590	0.97	1.07	0.375-1.41 0.548-1.24	0.440	10107401 EPA625	151010 GC-MS-SIM	Acceptable

Performance Summary

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Product: PAH-HPLC **Lot Number: 44633** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Benzo(g,h,i)perylene	N	WP	5590	0.437	1.07	0.375-1.41 0.548-1.24	2.64	10187607 EPA8310	251020 HPLC-UV	**Check for Error**
Benzo(k)fluoranthene	N	WP	5600	0.71	1.16	0.630-1.42 0.761-1.29	2.39	10107401 EPA625	151010 GC-MS-SIM	**Check for Error**
Benzo(k)fluoranthene	N	WP	5600	0.498	1.16	0.630-1.42 0.761-1.29	4.01	10187607 EPA8310	251020 HPLC-UV	**Not Acceptable**
Chrysene	N	WP	5855	<0.16				10107401 EPA625	151010 GC-MS-SIM	Acceptable
Chrysene	N	WP	5855	<0.16				10187607 EPA8310	251020 HPLC-UV	Acceptable
Dibenz(a,h) anthracene	N	WP	5895	<0.10				10107401 EPA625	151010 GC-MS-SIM	Acceptable
Dibenz(a,h) anthracene	N	WP	5895	<0.1				10187607 EPA8310	251020 HPLC-UV	Acceptable
Fluoranthene	N	WP	6265	0.98	1.39	0.815-1.68 0.958-1.53	1.85	10107401 EPA625	151010 GC-MS-SIM	Acceptable
Fluoranthene	N	WP	6265	0.576	1.39	0.815-1.68 0.958-1.53	4.66	10187607 EPA8310	251020 HPLC-UV	**Not Acceptable**
Fluorene	N	WP	6270	1.69	2.64	1.01-3.32 1.40-2.93	1.24	10107401 EPA625	151010 GC-MS-SIM	Acceptable
Fluorene	N	WP	6270	1.05	2.64	1.01-3.32 1.40-2.93	2.91	10187607 EPA8310	251020 HPLC-UV	**Check for Error**
Indeno (1,2,3-cd) pyrene	N	WP	6315	<0.25				10107401 EPA625	151010 GC-MS-SIM	Acceptable
Indeno (1,2,3-cd) pyrene	N	WP	6315	<0.25				10187607 EPA8310	251020 HPLC-UV	Acceptable

Performance Summary

APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

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Product: PAH-HPLC **Lot Number: 44633** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Naphthalene	N	WP	5005	2.37	3.66	1.06-4.39 1.62-3.84	0.642	10107401 EPA625	151010 GC-MS-SIM	Acceptable
Naphthalene	N	WP	5005	1.57	3.66	1.06-4.39 1.62-3.84	2.08	10187607 EPA8310	251020 HPLC-UV	**Check for Error**
Phenanthrene	N	WP	6615	0.45	0.641	0.318-0.836 0.404-0.749	1.47	10107401 EPA625	151010 GC-MS-SIM	Acceptable
Phenanthrene	N	WP	6615	0.273	0.641	0.318-0.836 0.404-0.749	3.52	10187607 EPA8310	251020 HPLC-UV	**Not Acceptable**
Pyrene	N	WP	6665	0.70	1.05	0.611-1.27 0.721-1.16	2.19	10107401 EPA625	151010 GC-MS-SIM	**Check for Error**
Pyrene	N	WP	6665	0.494	1.05	0.611-1.27 0.721-1.16	4.06	10187607 EPA8310	251020 HPLC-UV	**Not Acceptable**

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Product: PCBs in Oil

Lot Number: 44713-44714

Units: mg/kg

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aroclor 1016/1242 in Oil Sample 1	NE	WP	8880	<1.0				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1016/1242 in Oil Sample 2	NE	WP	8880	40.2	42.9	8.02-57.2	0.926	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1254 in Oil Sample 1	NE	WP	8905	41.3	37.4	16.2-49.0 5.66-54.8	1.35	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1254 in Oil Sample 2	NE	WP	8905	<1.0		13.9-46.6		10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1260 in Oil Sample 1	NE	WP	8910	<1.0				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1260 in Oil Sample 2	NE	WP	8910	<1.0				10103603 EPA608	152010 GC-ECD	Acceptable

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Product: PCBs in Water **Lot Number: 44722-44723** **APG+ Lot Number: 44724** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aroclor 1016 Sample 1	NE	WP	8880	<0.1				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1016 Sample 2	NE	WP	8880	<0.1				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1221 Sample 1	N	WP	8885	<0.1				10103603 EPA 608	152010 GC-ECD	Acceptable
Aroclor 1221 Sample 2		WP	8885	<0.1				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1232 Sample 1	NE	WP	8890	<0.1				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1232 Sample 2	NE	WP	8890	<0.1				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1242 Sample 1	NE	WP	8895	<0.1				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1242 Sample 2	NE	WP	8895	<0.1				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1248 Sample 1	NE	WP	8900	<0.1				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1248 Sample 2	NE	WP	8900	3.69	4.13	1.71-5.81 2.39-5.13	0.103	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1254 Sample 1	NE	WP	8905	2.86	3.20	1.42-4.32 1.91-3.84	0.0270	10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1254 Sample 2	NE	WP	8905	<0.1				10103603 EPA608	152010 GC-ECD	Acceptable
Aroclor 1260 Sample 1	NE	WP	8910	<0.1				10103603 EPA608	152010 GC-ECD	Acceptable

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EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**12/05/2005**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**10/03/2005**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**11/16/2005**

Product: PCBs in Water **Lot Number: 44722-44723** **APG+ Lot Number: 44724** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aroclor 1260 Sample 2	NE	WP	8910	<0.1				10103603 EPA608	152010 GC-ECD	Acceptable

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Product: Pesticides (NELAC) **Lot Number: 44694** **APG+ Lot Number: 44695** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
alpha-BHC	N	WP	7110	4.43	7.02	3.03-9.58 4.12-8.49	1.72	10103603 EPA608	152010 GC-ECD	Acceptable
alpha-Chlordane	N	WP	7240	3.95	5.82	2.61-7.87 3.49-6.99	1.48	10103603 EPA608		Acceptable
beta-BHC	N	WP	7115	4.96	9.51	3.96-12.9 5.45-11.4	2.33	10103603 EPA608	152010 GC-ECD	**Check for Error**
delta-BHC	N	WP	7105	8.21	11.9	4.55-16.6 6.56-14.6	1.18	10103603 EPA608	152010 GC-ECD	Acceptable
Endosulfan I	N	WP	7510	3.13	5.26	1.27-7.35 2.29-6.34	1.17	10103603 EPA608	152010 GC-ECD	Acceptable
Endosulfan II	N	WP	7515	10.1	13.0	4.01-17.2 6.21-15.0	0.234	10103603 EPA608	152010 GC-ECD	Acceptable
Endosulfan sulfate	N	WP	7520	5.90	7.60	2.85-11.1 4.22-9.72	0.779	10103603 EPA608	152010 GC-ECD	Acceptable
Endrin	N	WP	7540	6.40	8.73	3.11-13.0 4.76-11.4	1.00	10103603 EPA608	152010 GC-ECD	Acceptable
Endrin aldehyde	N	WP	7530	5.45	7.25	2.02-11.1 3.54-9.59	0.736	10103603 EPA608	152010 GC-ECD	Acceptable
Endrin Ketone	N	WP	7535	5.70	7.36	4.05-10.7 5.15-9.57	1.50	10103603 EPA608		Acceptable
gamma-BHC	N	WP	7120	3.11	5.18	2.09-7.20 2.94-6.34	1.80	10103603 EPA608	152010 GC-ECD	Acceptable
gamma-Chlordane	N	WP	7245	3.51	5.37	2.22-7.32 3.07-6.47	1.48	10103603 EPA608		Acceptable
Methoxychlor	N	WP	7810	4.39	5.44	1.38-8.76 2.61-7.53	0.549	10103603 EPA608	152010 GC-ECD	Acceptable

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Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/03/2005
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Product: Pesticides (WP) **Lot Number: 44691** **APG+ Lot Number: 44695** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
4,4'-DDD	NE	WP	7355	3.34	3.65	1.40-5.31 2.05-4.66	0.0211	10103603 EPA608	152010 GC-ECD	Acceptable
4,4'-DDE	NE	WP	7360	2.89	3.06	1.32-4.07 1.78-3.62	0.417	10103603 EPA608	152010 GC-ECD	Acceptable
4,4'-DDT	NE	WP	7365	4.83	5.26	2.96-6.51 3.55-5.92	0.162	10103603 EPA608	152010 GC-ECD	Acceptable
Aldrin	NE	WP	7025	0.620	0.711	0.227-1.02 0.359-0.886	0.0176	10103603 EPA608	152010 GC-ECD	Acceptable
Dieldrin	NE	WP	7470	1.80	1.81	0.835-2.54 1.12-2.26	0.395	10103603 EPA608	152010 GC-ECD	Acceptable
Heptachlor	NE	WP	7685	1.65	2.26	0.737-3.16 1.14-2.76	0.738	10103603 EPA608	152010 GC-ECD	Acceptable
Heptachlor expoxide	NE	WP	7690	1.91	2.14	1.01-3.07 1.35-2.72	0.368	10103603 EPA608	152010 GC-ECD	Acceptable

Product: pH **Lot Number: 44586** **APG+ Lot Number: 44878** **Units: Units**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
pH	NE	WP	1900	6.21	6.19	5.99-6.39 6.06-6.32	0.300	10008205 EPA150.1	352010 POT	Acceptable

Product: Settleable Solids **Lot Number: 44999** **Units: ml/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Settleable Solids	N	WP	1965	19.5	24.3	18.9-31.3 21.0-29.2	2.72	10010603 EPA160.5		Check for Error

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Primary AA E-10254	15130 South Keeler	QA Director	Study Shipment Date 10/03/2005
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Product: Solids — **Lot Number: 103591** — **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range / Warn Range	ZScore	Method Code / Method Desc.	Technology Code / Technology Desc.	Evaluation
Total Dissolved Solids		WP	1705	135	156	111-201 / 126-186	1.38	10009004 / EPA160.1	201010 / GRAV	Acceptable
Total Solids	N	WP	1950	196	223	186-258 / 198-246	2.17	10009800 / EPA160.3	201010 / GRAV	**Check for Error**
Total Suspended Solids	NE	WP	1960	61.0	67.4	54.1-75.7 / 57.7-72.1	1.09	10009402 / EPA160.2	201010 / GRAV	Acceptable

Product: Sulfide — **Lot Number: 44766** — **APG+ Lot Number: 44767** — **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range / Warn Range	ZScore	Method Code / Method Desc.	Technology Code / Technology Desc.	Evaluation
Sulfide	N	WP	2005	4.54	5.64	2.44-8.20 / 3.40-7.24	0.811	10074405 / EPA376.2		Acceptable

Product: Total Chlordane — **Lot Number: 44562** — **APG+ Lot Number: 44599** — **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range / Warn Range	ZScore	Method Code / Method Desc.	Technology Code / Technology Desc.	Evaluation
Total Chlordane	NE	WP	7250	12.9	12.0	4.50-17.3 / 6.64-15.2	0.922	10103603 / EPA608	152010 / GC-ECD	Acceptable

Product: Total Cyanide — **Lot Number: 44612** — **APG+ Lot Number: 44613** — **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range / Warn Range	ZScore	Method Code / Method Desc.	Technology Code / Technology Desc.	Evaluation
Total Cyanide	NE	WP	1645	0.600	0.553	0.331-0.778 / 0.406-0.703	0.613	10060205 / EPA335.2	401010 / COLOR	Acceptable

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Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/03/2005
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Product: Total Phenolics Lot Number: 44610 APG+ Lot Number: 44611 Units: mg/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Phenolics	NE	WP	1905	1.31	2.36	0.863-2.60	1.46	10079604	401010	Acceptable
						1.15-2.31		EPA420.2	COLOR	

Product: Total Residual Chlorine Lot Number: 44728 APG+ Lot Number: 44729 Units: mg/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Residual Chlorine	NE	WP	1940	1.4	1.35	0.969-1.67	0.679	10059004	651010	Acceptable
						1.09-1.55		EPA330.4	TITR	

Product: Toxaphene Lot Number: 44600 APG+ Lot Number: 44601 Units: ug/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Toxaphene	N	WP	8250	65.3	70.3	7.03-127	0.0858	10103603	152010	Acceptable
						27.0-107		EPA608	GC-ECD	

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Product: Trace Metals **Lot Number:** 44736-44737 **APG+ Lot Number:** 44738-44739 **Units:** ug/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Aluminum	NE	WP	1000	1420	1460	1190-1710 1280-1630	0.372	10014401 EPA200.8	102010 ICP-MS	Acceptable
Aluminum	NE	WP	1000	1400	1460	1190-1710 1280-1630	0.601	10013408 EPA200.7	102015 ICP-AES	Acceptable
Antimony	NE	WP	1005	340	364	251-440 282-409	0.172	10014401 EPA200.8	102010 ICP-MS	Acceptable
Antimony	NE	WP	1005	398	364	251-440 282-409	1.66	10013408 EPA200.7	102015 ICP-AES	Acceptable
Arsenic	NE	WP	1010	150	122	98.4-146 106-138	3.56	10014401 EPA200.8	102010 ICP-MS	**Not Acceptable**
Arsenic	NE	WP	1010	117	122	98.4-146 106-138	0.637	10013408 EPA200.7	102015 ICP-AES	Acceptable
Barium	N	WP	1015	1370	1410	1220-1590 1290-1530	0.612	10014401 EPA200.8	102010 ICP-MS	Acceptable
Barium	N	WP	1015	1350	1410	1220-1590 1290-1530	0.940	10013408 EPA200.7	102015 ICP-AES	Acceptable
Beryllium	NE	WP	1020	506	515	438-582 462-558	0.156	10014401 EPA200.8	102010 ICP-MS	Acceptable
Beryllium	NE	WP	1020	487	515	438-582 462-558	0.949	10013408 EPA200.7	102015 ICP-AES	Acceptable
Boron	N	WP	1025	986	1000	825-1170 882-1110	0.167	10014401 EPA200.8	102010 ICP-MS	Acceptable
Boron	N	WP	1025	1020	1000	825-1170 882-1110	0.431	10013408 EPA200.7	102015 ICP-AES	Acceptable
Cadmium	NE	WP	1030	68.7	71.6	60.3-82.5 64.0-78.8	0.729	10014401 EPA200.8	102010 ICP-MS	Acceptable

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Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**10/03/2005**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**11/16/2005**

Product: Trace Metals **Lot Number: 44736-44737** **APG+ Lot Number: 44738-44739** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Cadmium	NE	WP	1030	75.4	71.6	60.3-82.5 64.0-78.8	1.08	10013408 EPA200.7	102015 ICP-AES	Acceptable
Chromium	NE	WP	1040	432	434	377-491 396-472	0.126	10014401 EPA200.8	102010 ICP-MS	Acceptable
Chromium	NE	WP	1040	451	434	377-491 396-472	0.873	10013408 EPA200.7	102015 ICP-AES	Acceptable
Cobalt	NE	WP	1050	131	136	118-153 124-147	0.809	10014401 EPA200.8	102010 ICP-MS	Acceptable
Cobalt	NE	WP	1050	138	136	118-153 124-147	0.384	10013408 EPA200.7	102015 ICP-AES	Acceptable
Copper	NE	WP	1055	123	123	109-138 114-133	0.0599	10014401 EPA200.8	102010 ICP-MS	Acceptable
Copper	NE	WP	1055	125	123	109-138 114-133	0.350	10013408 EPA200.7	102015 ICP-AES	Acceptable
Iron	NE	WP	1070	589	573	504-650 529-626	0.478	10013408 EPA200.7	102015 ICP-AES	Acceptable
Iron	NE	WP	1070	556	573	504-650 529-626	0.876	10014401 EPA200.8	102010 ICP-MS	Acceptable
Lead	NE	WP	1075	1100	1150	1010-1280 1060-1240	1.03	10014401 EPA200.8	102010 ICP-MS	Acceptable
Lead	NE	WP	1075	1200	1150	1010-1280 1060-1240	1.15	10013408 EPA200.7	102015 ICP-AES	Acceptable
Manganese	NE	WP	1090	1280	1320	1190-1470 1240-1420	1.07	10014401 EPA200.8	102010 ICP-MS	Acceptable
Manganese	NE	WP	1090	1370	1320	1190-1470 1240-1420	0.857	10013408 EPA200.7	102015 ICP-AES	Acceptable

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Product: Trace Metals **Lot Number: 44736-44737** **APG+ Lot Number: 44738-44739** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Mercury	NE	WP	1095	6.93	6.55	4.04-8.96 4.86-8.14	0.525	10036201 EPA245.1	101010 CVAAS	Acceptable
Mercury	NE	WP	1095	5.37	6.55	4.04-8.96 4.86-8.14	1.38	10014401 EPA200.8	102010 ICP-MS	Acceptable
Molybdenum	NE	WP	1100	443	442	374-505 396-483	0.148	10013408 EPA200.7	102015 ICP-AES	Acceptable
Molybdenum	NE	WP	1100	415	442	374-505 396-483	1.14	10014401 EPA200.8	102010 ICP-MS	Acceptable
Nickel	NE	WP	1105	453	466	417-523 435-505	0.977	10014401 EPA200.8	102010 ICP-MS	Acceptable
Nickel	NE	WP	1105	492	466	417-523 435-505	1.24	10013408 EPA200.7	102015 ICP-AES	Acceptable
Selenium	NE	WP	1140	1090	1100	875-1270 941-1210	0.243	10014401 EPA200.8	102010 ICP-MS	Acceptable
Selenium	NE	WP	1140	1130	1100	875-1270 941-1210	0.846	10013408 EPA200.7	102015 ICP-AES	Acceptable
Silver	NE	WP	1150	383	359	308-411 325-394	1.36	10013408 EPA200.7	102015 ICP-AES	Acceptable
Silver	NE	WP	1150	357	359	308-411 325-394	0.153	10014401 EPA200.8	102010 ICP-MS	Acceptable
Strontium	NE	WP	1160	88.8	94.2	79.7-109 84.5-104	1.12	10014401 EPA200.8	102010 ICP-MS	Acceptable
Strontium	NE	WP	1160	89.9	94.2	79.7-109 84.5-104	0.889	10013408 EPA200.7	102015 ICP-AES	Acceptable
Thallium	NE	WP	1165	285	313	240-385 264-360	1.13	10014401 EPA200.8	102010 ICP-MS	Acceptable

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Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**10/03/2005**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**11/16/2005**

Product: Trace Metals **Lot Number: 44736-44737** **APG+ Lot Number: 44738-44739** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Thallium	NE	WP	1165	263	313	240-385 264-360	2.04	10013408 EPA200.7	102015 ICP-AES	**Check for Error**
Tin	N	WP	1175	2690	2750	2170-3350 2360-3150	0.341	10014401 EPA200.8	102010 ICP-MS	Acceptable
Tin	N	WP	1175	2870	2750	2170-3350 2360-3150	0.575	10013408 EPA200.7	102015 ICP-AES	Acceptable
Titanium	NE	WP	1180	134	136	116-154 123-148	0.172	10013408 EPA200.7	102015 ICP-AES	Acceptable
Titanium	NE	WP	1180	124	136	116-154 123-148	1.76	10014401 EPA200.8	102010 ICP-MS	Acceptable
Vanadium	NE	WP	1185	226	238	208-267 218-257	1.16	10014401 EPA200.8	102010 ICP-MS	Acceptable
Vanadium	NE	WP	1185	232	238	208-267 218-257	0.552	10013408 EPA200.7	102015 ICP-AES	Acceptable
Zinc	NE	WP	1190	1240	1280	1100-1470 1160-1410	0.721	10014401 EPA200.8	102010 ICP-MS	Acceptable
Zinc	NE	WP	1190	1360	1280	1100-1470 1160-1410	1.25	10013408 EPA200.7	102015 ICP-AES	Acceptable

Product: Turbidity **Lot Number: 44679** **APG+ Lot Number: 44680** **Units: NTU**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Turbidity	N	WP	2055	1.43	1.71	1.25-2.23 1.41-2.06	1.88	10011402 EPA180.1		Acceptable

Performance Summary

3655

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		WP October 2005 WP #5010
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	Study Print Date	12/05/2005
Primary AA	E-10254	15130 South Keeler	QA Director	Study Shipment Date	10/03/2005
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	Study Closing Date	11/16/2005

Product: Uranium **Lot Number: 44565** **APG+ Lot Number: 44566** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Uranium	X	WP	3035	124	130	110-141 115-136	0.246	10014401 EPA200.8		Acceptable

Performance Summary

3655

APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP October 2005 WP #5010
Study Print Date 12/05/2005
Study Shipment Date 10/03/2005
Study Closing Date 11/16/2005

Product: Volatile Aromatics **Lot Number: 43596** **APG+ Lot Number: 44735** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
1,2-Dichlorobenzene	NE	WP	4610	43.0	42.5	29.2-55.4 33.6-51.0	0.158	10107207 EPA624	151010 GC-MS	Acceptable
1,2-Dichlorobenzene	NE	WP	4610	41.3	42.5	29.2-55.4 33.6-51.0	0.232	10102202 EPA602		Acceptable
1,3-Dichlorobenzene	NE	WP	4615	101	89.2	60.8-113 69.6-105	1.59	10107207 EPA624	151010 GC-MS	Acceptable
1,3-Dichlorobenzene	NE	WP	4615	88.9	89.2	60.8-113 69.6-105	0.208	10102202 EPA602		Acceptable
1,4-Dichlorobenzene	NE	WP	4620	39.7	39.6	26.6-50.4 30.5-46.4	0.313	10107207 EPA624	151010 GC-MS	Acceptable
1,4-Dichlorobenzene	NE	WP	4620	34.2	39.6	26.6-50.4 30.5-46.4	1.07	10102202 EPA602		Acceptable
Benzene	NE	WP	4375	39.8	38.4	27.3-49.3 31.0-45.6	0.410	10107207 EPA624	151010 GC-MS	Acceptable
Benzene	NE	WP	4375	32.4	38.4	27.3-49.3 31.0-45.6	1.61	10102202 EPA602		Acceptable
Ethylbenzene	NE	WP	4765	15.8	15.2	10.1-20.1 11.8-18.4	0.414	10107207 EPA624	151010 GC-MS	Acceptable
Ethylbenzene	NE	WP	4765	16.1	15.2	10.1-20.1 11.8-18.4	0.595	10102202 EPA602		Acceptable
Toluene	NE	WP	5140	66.9	63.7	44.2-79.8 50.1-73.8	0.829	10107207 EPA624	151010 GC-MS	Acceptable
Toluene	NE	WP	5140	54.0	63.7	44.2-79.8 50.1-73.8	1.35	10102202 EPA602		Acceptable
Total Xylenes	N	WP	5260	70.9	69.0	39.0-94.4 48.2-85.2	0.455	10107207 EPA624	151010 GC-MS	Acceptable

Performance Summary

APG Customer	3655	Laboratory Address	Nelac Point Of Contact		**WP October 2005 WP #5010**
EPA Lab Code	KS00902	Analytical Management Lab	Tenkasi Viswanathan	**Study Print Date**	**12/05/2005**
Primary AA	E-10254	15130 South Keeler	QA Director	**Study Shipment Date**	**10/03/2005**
APG NIST/NVLAP	200384-0	Olathe, KS 66062	913-829-0101 x26	**Study Closing Date**	**11/16/2005**

Product: Volatile Aromatics **Lot Number: 43596** **APG+ Lot Number: 44735** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Total Xylenes	N	WP	5260	68.4	69.0	39.0-94.4 48.2-85.2	0.185	10102202 EPA602		Acceptable

Product: Volatile Solids **Lot Number: 44711** **Units: mg/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Volatile solids	N	WP	1970		201	152-236 166-222				Not Reported

Performance Summary

APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

3655
WP October 2005 WP #5010
Study Print Date 12/05/2005
Study Shipment Date 10/03/2005
Study Closing Date 11/16/2005

Product: Volatiles

Lot Number: 44733 APG+ Lot Number: 44927 Units: ug/L

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
1,1,1,2-Tetrachloroethane		WP	5105	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
1,1,1-Trichloroethane	NE	WP	5160	18.3	18.0	11.4-24.5 13.6-22.3	0.158	10107207 EPA624	151010 GC-MS	Acceptable
1,1,2,2-Tetrachloroethane	N	WP	5110	21.1	20.8	10.5-33.0 14.2-29.3	0.173	10107207 EPA624	151010 GC-MS	Acceptable
1,1,2-Trichloroethane	N	WP	5165	132	124	85.7-160 98.0-147	0.751	10107207 EPA624	151010 GC-MS	Acceptable
1,1-Dichloroethane	N	WP	4630	91.8	87.5	60.4-120 70.3-110	0.160	10107207 EPA624	151010 GC-MS	Acceptable
1,1-Dichloroethene	N	WP	4640	28.1	26.9	14.2-39.3 18.4-35.1	0.327	10107207 EPA624	151010 GC-MS	Acceptable
1,2-Dichloroethane	NE	WP	4635	116	116	80.6-151 92.4-140	0.00048	10107207 EPA624	151010 GC-MS	Acceptable
1,2-Dichloropropane	N	WP	4655	130	119	78.5-156 91.5-143	0.979	10107207 EPA624	151010 GC-MS	Acceptable
2-Butanone		WP	4410	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
2-Chloroethyl vinyl ether		WP	4500	<5.0				10107207 EPA624	151010 GC-MS	Acceptable
2-Hexanone	N	WP	4860	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
4-Methyl-2-pentanone (MIBK)	N	WP	4995	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
Acetone		WP	4315	<2.0				10107207 EPA624	151010 GC-MS	Acceptable

Performance Summary

3655

APG Customer 3655
EPA Lab Code KS00902
Primary AA E-10254
APG NIST/NVLAP 200384-0

Laboratory Address
Analytical Management Lab
15130 South Keeler
Olathe, KS 66062

Nelac Point Of Contact
Tenkasi Viswanathan
QA Director
913-829-0101 x26

WP October 2005 WP #5010
Study Print Date 12/05/2005
Study Shipment Date 10/03/2005
Study Closing Date 11/16/2005

Product: Volatiles **Lot Number: 44733** **APG+ Lot Number: 44927** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
Acetonitrile		WP	4320	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
Acrolein		WP	4325	13.5				10107207 EPA624	151010 GC-MS	**Not Acceptable**
Acrylonitrile		WP	4340	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
Bromodichloromethane	NE	WP	4395	41.5	39.3	27.6-53.0 31.8-48.8	0.286	10107207 EPA624	151010 GC-MS	Acceptable
Bromoform	NE	WP	4400	21.3	18.5	10.7-24.9 13.1-22.5	1.47	10107207 EPA624	151010 GC-MS	Acceptable
Bromomethane	N	WP	4950	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
Carbon disulfide		WP	4450	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
Carbon tetrachloride	NE	WP	4455	113	101	54.8-137 68.6-124	1.23	10107207 EPA624	151010 GC-MS	Acceptable
Chlorobenzene	NE	WP	4475	12.0	12.2	8.69-15.8 9.88-14.6	0.205	10107207 EPA624	151010 GC-MS	Acceptable
Chloroethane	N	WP	4485	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
Chloroform	NE	WP	4505	22.2	20.9	14.3-28.0 16.6-25.7	0.465	10107207 EPA624	151010 GC-MS	Acceptable
Chloromethane	N	WP	4960	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
cis-1,2-Dichloroethene	N	WP	4645	<2.0				10107207 EPA624	151010 GC-MS	Acceptable

Performance Summary

3655

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EPA Lab Code KS00902
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Olathe, KS 66062

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913-829-0101 x26

WP October 2005 WP #5010
Study Print Date 12/05/2005
Study Shipment Date 10/03/2005
Study Closing Date 11/16/2005

Product: Volatiles **Lot Number: 44733** **APG+ Lot Number: 44927** **Units: ug/L**

Analyte		Product Level	NELAC Code	Reported Value	Assigned Value	Accept Range Warn Range	ZScore	Method Code Method Desc.	Technology Code Technology Desc.	Evaluation
cis-1,3-Dichloropropene	N	WP	4680	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
Dibromochloromethane	NE	WP	4575	79.6	74.7	51.2-99.2 59.2-91.2	0.551	10107207 EPA624	151010 GC-MS	Acceptable
Methyl-t-butylether (MTBE)	N	WP	5000	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
Methylene chloride (Dichloromethane)	NE	WP	4975	72.4	73.0	44.7-101 54.2-91.9	0.0700	10107207 EPA624	151010 GC-MS	Acceptable
Styrene	N	WP	5100	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
Tetrachloroethene	NE	WP	5115	97.9	89.3	50.7-116 61.7-106	1.31	10107207 EPA624	151010 GC-MS	Acceptable
trans-1,2-Dichloroethene	N	WP	4700	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
trans-1,3-Dichloropropene	N	WP	4685	40.7	40.2	25.4-54.0 30.2-49.2	0.215	10107207 EPA624	151010 GC-MS	Acceptable
Trichloroethene	NE	WP	5170	91.7	84.1	53.5-109 62.8-100	1.11	10107207 EPA624	151010 GC-MS	Acceptable
Trichlorofluoromethane	N	WP	5175	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
Vinyl acetate		WP	5225	<2.0				10107207 EPA624	151010 GC-MS	Acceptable
Vinyl Chloride	N	WP	5235	<2.0				10107207 EPA624	151010 GC-MS	Acceptable

SOIL-33 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 EPA ID: KS00902 State ID: Report Issued: 03/28/01

InterLaB™ SOIL Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Value for the Organic Proficiency Testing Standards is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation as applicable. The ERA Assigned Value for the Inorganic Proficiency Testing Standards is equal to the maximum amount of the parameter available in the standard by applicable EPA methodologies. All NELAC parameters not added to a standard are given an assigned Value of "0", per the guidance issued by the NELAC Board of Directors, on December 14, 2000. Non-NELAC parameters not added to a standard may be given an Assigned Value of less than a minimum verified concentration as determined in the background soil for applicable EPA methodologies.

The Acceptance Limits are established per the NELAC PT program criteria, state specific PT program requirements or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For a further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside the Acceptance Limits.
No Evaluation	=	Reported Value that can not be evaluated.

The Method Description is the method the laboratory reported to ERA.

D.L. equals the Detection Limit.

InterLaB™ SOIL Study Discussion:

ERA Soil Proficiency Testing Study 33 has been reviewed by ERA Senior Management and certified compliant with the requirements of the National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program, Chapter 2, June 2000.

Per the requirements of the NELAC Proficiency Testing Program, the SOIL-33 results were examined for any study anomalies. A full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the SOIL-33 standards met the acceptance criteria contained in the NELAC Proficiency Testing Program, Chapter 2, June 2000.

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's review of the data, no study anomolies were found and all Acceptance Limits for the InterLaB™ Soil study, SOIL-33, were calculated based on the NELAC, Proficiency Testing Program, Chapter 2, June 2000, as applicable.

The Soil Proficiency Testing Program study SOIL-33 report shall not be reproduced except in its entirety and without the full written permission of the participating laboratory. The report must not be used by the participating laboratory to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding Soil Study, SOIL-33, please contact Shawn Kassner, InterLaB™ Program Coordinator, or Curtis Wood, Quality Assurance Manager, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

ENVIRONMENTAL
RESOURCE ASSOCIATES®

Study: **SOIL33**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **ANALYTICAL MANAGEMENT LABS**

Report Type: **Complete**

Report Method: **Method A**

5540 Marshall St., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-33 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil						
Aluminum	mg/Kg	5852	6040	1270 - 10200	Acceptable	EPA 6010B
Antimony	mg/Kg	210	243	0.00 - 243	Acceptable	EPA 6010B
Arsenic	mg/Kg	188	184	103 - 243	Acceptable	EPA 6010B
Barium	mg/Kg	155	148	104 - 198	Acceptable	EPA 6010B
Beryllium	mg/Kg	61.7	69.5	42.0 - 82.4	Acceptable	EPA 6010B
Boron	mg/Kg		133	28.6 - 133		
Cadmium	mg/Kg	198	219	126 - 248	Acceptable	EPA 6010B
Calcium	mg/Kg	3382	3310	1970 - 4430	Acceptable	EPA 6010B
Chromium	mg/Kg	119	117	70.4 - 150	Acceptable	EPA 6010B
Cobalt	mg/Kg	51.5	70.6	34.5 - 70.6	Acceptable	EPA 6010B
Copper	mg/Kg	87.1	87.6	60.1 - 113	Acceptable	EPA 6010B
Iron	mg/Kg	10203	11800	330 - 21300	Acceptable	EPA 6010B
Lead	mg/Kg	104	110	64.4 - 150	Acceptable	EPA 6010B
Magnesium	mg/Kg	1908	1960	881 - 2940	Acceptable	EPA 6010B
Manganese	mg/Kg	370	382	230 - 496	Acceptable	EPA 6010B
Mercury	mg/Kg		28.6	14.1 - 44.7		
Molybdenum	mg/Kg	86.7	92.8	56.0 - 109	Acceptable	EPA 6010B
Nickel	mg/Kg	139	142	85.9 - 168	Acceptable	EPA 6010B
Potassium	mg/Kg	1886	1820	621 - 3100	Acceptable	EPA 6010B
Selenium	mg/Kg	91.5	95.9	51.5 - 124	Acceptable	EPA 6010B
Silver	mg/Kg	72.9	207	0.00 - 507	Acceptable	EPA 6010B
Sodium	mg/Kg	223	218	0.00 - 573	Acceptable	EPA 6010B
Strontium	mg/Kg		182	117 - 225		
Thallium	mg/Kg	158	166	85.5 - 215	Acceptable	EPA 6010B
Tin	mg/Kg		167	55.0 - 167		
Titanium	mg/Kg		282	0.00 - 669		
Vanadium	mg/Kg	76.2	84.2	39.9 - 113	Acceptable	EPA 6010B
Zinc	mg/Kg	396	414	261 - 493	Acceptable	EPA 6010B
Cyanide in Soil						
Cyanide, total	mg/Kg	136	277	0.00 - 461	Acceptable	EPA 335.2 9010/901
Reactive Cyanide	mg/Kg		< 25.0			

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SOIL-33 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Hexavalent Chromium in Soil						
Chromium VI	mg/Kg	96.9	177	47.5 - 225	Acceptable	EPA 3060A

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-33 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
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EPA ID: KS00902 **Report Issued: 03/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil						
Acenaphthene	µg/Kg	3037	3210	730 - 4210	Acceptable	EPA 8270C
Acenaphthylene	µg/Kg	2138	2650	422 - 3040	Acceptable	EPA 8270C
Aniline	µg/Kg		0.00			
Anthracene	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
Benzidine	µg/Kg		0.00			
Benzoic acid	µg/Kg		0.00			
Benzo(a)anthracene	µg/Kg	3330	4170	998 - 5820	Acceptable	EPA 8270C
Benzo(b)fluoranthene	µg/Kg	5293	6310	1590 - 8850	Acceptable	EPA 8270C
Benzo(k)fluoranthene	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
Benzo(g,h,i)perylene	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
Benzo(a)pyrene	µg/Kg	3678	5780	1010 - 6730	Acceptable	EPA 8270C
Benzyl alcohol	µg/Kg		0.00			
4-Bromophenyl-phenylether	µg/Kg	5309	6700	1540 - 9400	Acceptable	EPA 8270C
Butylbenzylphthalate	µg/Kg	8150	9450	1880 - 13900	Acceptable	EPA 8270C
Carbazole	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
4-Chloroaniline	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
bis(2-Chloroethoxy)methane	µg/Kg	5101	5820	660 - 7980	Acceptable	EPA 8270C
bis(2-Chloroethyl)ether	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
bis(2-Chloroisopropyl)ether	µg/Kg	< 66.7	1530	0.00 - 2550	Acceptable	EPA 8270C
4-Chloro-3-methylphenol	µg/Kg	4745	7390	1110 - 8250	Acceptable	EPA 8270C
1-Chloronaphthalene	µg/Kg		0.00			
2-Chloronaphthalene	µg/Kg	2292	3380	745 - 4320	Acceptable	EPA 8270C
2-Chlorophenol	µg/Kg	2786	4360	303 - 4740	Acceptable	EPA 8270C
4-Chlorophenyl-phenylether	µg/Kg	3498	3710	679 - 5280	Acceptable	EPA 8270C
Chrysene	µg/Kg	3023	3700	1020 - 5180	Acceptable	EPA 8270C
Dibenz(a,h)anthracene	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
Dibenzofuran	µg/Kg	1795	2080	472 - 2910	Acceptable	EPA 8270C
Di-n-butylphthalate	µg/Kg	4874	4420	1110 - 6710	Acceptable	EPA 8270C
1,2-Dichlorobenzene	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
1,3-Dichlorobenzene	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
1,4-Dichlorobenzene	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
3,3'-Dichlorobenzidine	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
2,4-Dichlorophenol	µg/Kg	2365	4080	553 - 4270	Acceptable	EPA 8270C
2,6-Dichlorophenol	µg/Kg		0.00			
Diethylphthalate	µg/Kg	4623	5890	1190 - 8150	Acceptable	EPA 8270C
2,4-Dimethylphenol	µg/Kg	< 66.7	6790	0.00 - 6790	Acceptable	EPA 8270C
Dimethylphthalate	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
4,6-Dinitro-2-methylphenol	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
2,4-Dinitrophenol	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
2,4-Dinitrotoluene	µg/Kg	5474	6720	1290 - 9030	Acceptable	EPA 8270C
2,6-Dinitrotoluene	µg/Kg	5864	7250	2120 - 9320	Acceptable	EPA 8270C

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ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)						
Di-n-octylphthalate	µg/Kg	7438	9280	10.0 - 15500	Acceptable	EPA 8270C
bis(2-Ethylhexyl)phthalate	µg/Kg	3619	3770	825 - 5780	Acceptable	EPA 8270C
Fluoranthene	µg/Kg	5528	5960	1440 - 8400	Acceptable	EPA 8270C
Fluorene	µg/Kg	4145	4430	887 - 6170	Acceptable	EPA 8270C
Hexachlorobenzene	µg/Kg	2436	2880	780 - 3960	Acceptable	EPA 8270C
Hexachlorobutadiene	µg/Kg	2263	2810	168 - 3610	Acceptable	EPA 8270C
Hexachlorocyclopentadiene	µg/Kg	72.7	5620	0.00 - 5620	Acceptable	EPA 8270C
Hexachloroethane	µg/Kg	2470	3600	0.00 - 4340	Acceptable	EPA 8270C
Indeno(1,2,3-cd)pyrene	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
Isophorone	µg/Kg	2547	2660	466 - 3630	Acceptable	EPA 8270C
2-Methylnaphthalene	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
2-Methylphenol	µg/Kg	1519	5040	0.00 - 5040	Acceptable	EPA 8270C
3-Methylphenol	µg/Kg		0.00			
4-Methylphenol	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
Naphthalene	µg/Kg	5168	6010	820 - 7420	Acceptable	EPA 8270C
2-Nitroaniline	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
3-Nitroaniline	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
4-Nitroaniline	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
Nitrobenzene	µg/Kg	4271	6000	910 - 7210	Acceptable	EPA 8270C
2-Nitrophenol	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
3-Nitrophenol	µg/Kg		0.00			
4-Nitrophenol	µg/Kg	4983	9820	0.00 - 12000	Acceptable	EPA 8270C
N-Nitrosodimethylamine	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
N-Nitrosodiphenylamine	µg/Kg		0.00			
N-Nitroso-di-n-propylamine	µg/Kg	14497	7290	610 - 9730	Not Acceptable	EPA 8270C
Pentachlorophenol	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
Phenanthrene	µg/Kg	2365	2450	646 - 3570	Acceptable	EPA 8270C
Phenol	µg/Kg	8878	12100	880 - 13500	Acceptable	EPA 8270C
Pyrene	µg/Kg	5528	6710	1410 - 9630	Acceptable	EPA 8270C
Pyridine	µg/Kg		0.00			
1,2,4-Trichlorobenzene	µg/Kg		3450	275 - 4330		
2,4,5-Trichlorophenol	µg/Kg	2105	3510	414 - 3970	Acceptable	EPA 8270C
2,4,6-Trichlorophenol	µg/Kg	2100	3550	559 - 3720	Acceptable	EPA 8270C
Additional Base / Neutrals and Acids in Soil						
2-Amino-1-methylbenzene (o-touluidine)	µg/Kg		0.00			
Benzotrichloride	µg/Kg		0.00			
Benzyl chloride	µg/Kg		0.00			
bis(2-methoxyethyl)phthalate	µg/Kg		0.00			
2-Chlorophenyl-4-nitrophenylether	µg/Kg		0.00			
3-Chlorophenyl-4-nitrophenylether	µg/Kg		0.00			
4-Chlorophenyl-4-nitrophenylether	µg/Kg		0.00			

NVLAP® Lab code: 200386-0

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil (Cont.)						
Diamylphthalate	µg/Kg		0.00			
2,4-Dibromophenyl-4-nitrophenylether	µg/Kg		0.00			
2,4-Dichlorophenyl-3-methyl-4-nitrophenylether	µg/Kg		0.00			
2,3-Dichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,4-Dichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,5-Dichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,6-Dichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
3,4-Dichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
3,5-Dichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
Dicyclohexylphthalate	µg/Kg		0.00			
4-Nitrophenylphenylether	µg/Kg		0.00			
Dinonylphthalate	µg/Kg		0.00			
Hexachlorocyclohexane	µg/Kg		0.00			
Hexyl-2-ethylhexylphthalate	µg/Kg		0.00			
Maleic anhydride	µg/Kg		0.00			
Pentachlorobenzene	µg/Kg		0.00			
Pentachlorohexane	µg/Kg		0.00			
1,2,3,4-Tetrachlorobenzene	µg/Kg		0.00			
1,2,3,5-Tetrachlorobenzene	µg/Kg		0.00			
1,2,4,5-Tetrachlorobenzene	µg/Kg		0.00			
2,3,4,5-Tetrachlorophenol	µg/Kg		0.00			
2,3,4,6-Tetrachlorophenol	µg/Kg		0.00			
2,3,5,6-Tetrachlorophenol	µg/Kg		0.00			
1,2,3-Trichlorobenzene	µg/Kg		0.00			
1,3,5-Trichlorobenzene	µg/Kg		0.00			
2,3,4-Trichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,3,5-Trichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,3,6-Trichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,4,5-Trichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,4,6-Trichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
3,4,5-Trichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
Pesticides in Soil						
Aldrin	µg/Kg		151	69.5 - 205		
alpha-BHC	µg/Kg		128	55.2 - 173		
beta-BHC	µg/Kg		265	95.9 - 346		
delta-BHC	µg/Kg		341	143 - 453		
gamma-BHC(Lindane)	µg/Kg		107	46.9 - 146		
alpha-Chlordane	µg/Kg		148	22.2 - 236		
gamma-Chlordane	µg/Kg		0.00			
4,4'-DDD	µg/Kg		96.2	32.5 - 160		
4,4'-DDE	µg/Kg		232	83.5 - 339		

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Pesticides in Soil (Cont.)						
4,4'-DDT	µg/Kg		193	63.3 - 303		
Dieldrin	µg/Kg		319	140 - 446		
Endrin	µg/Kg		140	58.5 - 218		
Endrin aldehyde	µg/Kg		0.00			
Endrin ketone	µg/Kg		0.00			
Endosulfan I	µg/Kg		317	125 - 413		
Endosulfan II	µg/Kg		217	67.5 - 233		
Endosulfan sulfate	µg/Kg		279	85.4 - 417		
Heptachlor	µg/Kg		321	116 - 438		
Heptachlor epoxide	µg/Kg		110	49.4 - 171		
Methoxychlor	µg/Kg		412	13.0 - 721		
Chlordane in Soil						
Chlordane, Technical	µg/Kg	554	429	61.0 - 631	Acceptable	EPA 8081A
Toxaphene in Soil						
Toxaphene	µg/Kg	261	215	0.00 - 419	Acceptable	EPA 8081A
Volatiles in Soil						
Acetone	µg/Kg	110	61.3	0.00 - 324	Acceptable	EPA 8260B
Acetonitrile	µg/Kg		0.00			
Acrolein	µg/Kg		0.00			
Benzene	µg/Kg	75.2	75.8	45.9 - 117	Acceptable	EPA 8260B
Bromodichloromethane	µg/Kg	135	148	75.0 - 231	Acceptable	EPA 8260B
Bromoform	µg/Kg	89.4	88.5	41.4 - 147	Acceptable	EPA 8260B
Bromomethane	µg/Kg	< 10	0.00		Acceptable	EPA 8260B
2-Butanone (MEK)	µg/Kg	< 10	19.7	0.00 - 75.8	Acceptable	EPA 8260B
Carbon disulfide	µg/Kg	< 10	0.00		Acceptable	EPA 8260B
Carbon tetrachloride	µg/Kg	77.9	85.4	35.5 - 141	Acceptable	EPA 8260B
Chlorobenzene	µg/Kg	92.3	103	49.2 - 155	Acceptable	EPA 8260B
Chlorodibromomethane	µg/Kg	137	141	74.9 - 217	Acceptable	EPA 8260B
Chloroethane	µg/Kg	< 10	0.00		Acceptable	EPA 8260B
2-Chloroethylvinylether	µg/Kg		0.00			
Chloroform	µg/Kg	113	127	70.8 - 181	Acceptable	EPA 8260B
Chloromethane	µg/Kg	< 10	0.00		Acceptable	EPA 8260B
1,2-Dibromo-3-chloropropane	µg/Kg		0.00			
1,2-Dibromoethane (EDB)	µg/Kg	< 10	0.00		Acceptable	EPA 8260B
Dibromomethane	µg/Kg	< 10	0.00		Acceptable	EPA 8260B
1,2-Dichlorobenzene	µg/Kg	154	198	53.4 - 313	Acceptable	EPA 8260B
1,3-Dichlorobenzene	µg/Kg	108	141	46.1 - 216	Acceptable	EPA 8260B
1,4-Dichlorobenzene	µg/Kg	95	124	33.4 - 191	Acceptable	EPA 8260B
Dichlorodifluoromethane	µg/Kg	< 10	0.00		Acceptable	EPA 8260B
1,1-Dichloroethane	µg/Kg	31.7	37.9	21.4 - 55.9	Acceptable	EPA 8260B
1,2-Dichloroethane	µg/Kg	64.9	71.5	42.6 - 109	Acceptable	EPA 8260B

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SOIL-33 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
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ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)						
1,1-Dichloroethylene	µg/Kg	< 10	0.00		Acceptable	EPA 8260B
cis-1,2-Dichloroethylene	µg/Kg	< 10	0.00		Acceptable	EPA 8260B
trans-1,2-Dichloroethylene	µg/Kg	< 10	0.00		Acceptable	EPA 8260B
1,2-Dichloropropane	µg/Kg	< 10	0.00		Acceptable	EPA 8260B
cis-1,3-Dichloropropylene	µg/Kg	< 10	0.00		Acceptable	EPA 8260B
trans-1,3-Dichloropropylene	µg/Kg	< 10	0.00		Acceptable	EPA 8260B
Ethylbenzene	µg/Kg	68.6	72.2	36.0 - 116	Acceptable	EPA 8260B
2-Hexanone	µg/Kg	< 20	0.00		Acceptable	EPA 8260B
Methylene chloride	µg/Kg	81.0	91.3	40.3 - 147	Acceptable	EPA 8260B
4-Methyl-2-pentanone (MIBK)	µg/Kg	111	97.4	31.9 - 166	Acceptable	EPA 8260B
Styrene	µg/Kg	< 10	0.00		Acceptable	EPA 8260B
1,1,1,2-Tetrachloroethane	µg/Kg	51.7	55.6	29.0 - 89.0	Acceptable	EPA 8260B
1,1,2,2-Tetrachloroethane	µg/Kg	94.8	102	45.9 - 158	Acceptable	EPA 8260B
Tetrachloroethylene	µg/Kg	83.9	88.5	37.4 - 130	Acceptable	EPA 8260B
Toluene	µg/Kg	93.3	96.4	48.4 - 150	Acceptable	EPA 8260B
1,1,1-Trichloroethane	µg/Kg	160	170	99.0 - 243	Acceptable	EPA 8260B
1,1,2-Trichloroethane	µg/Kg	49.5	50.9	33.0 - 76.0	Acceptable	EPA 8260B
Trichloroethylene	µg/Kg	75.2	69.3	34.5 - 110	Acceptable	EPA 8260B
Trichlorofluoromethane	µg/Kg		0.00			
1,2,3-Trichloropropane	µg/Kg		64.9	20.0 - 109		
Vinyl acetate	µg/Kg		0.00			
Vinyl chloride	µg/Kg	< 10	0.00		Acceptable	EPA 8260B
Total, Xylenes	µg/Kg	164	176	65.8 - 290	Acceptable	EPA 8260B
Herbicides in Soil						
2,4-D	µg/Kg		390	0.00 - 822		
Dicamba	µg/Kg		212	0.00 - 392		
Disulfoton	µg/Kg		434	0.00 - 683		
2,4,5-T	µg/Kg		296	0.00 - 686		
2,4,5-TP (Silvex)	µg/Kg		139	0.00 - 284		
PAHs in Soil						
Acenaphthene	µg/Kg		< 100			
Acenaphthylene	µg/Kg		< 100			
Anthracene	µg/Kg		3280	0.00 - 4260		
Benzo(a)anthracene	µg/Kg		441	0.00 - 744		
Benzo(b)fluoranthene	µg/Kg		265	0.00 - 537		
Benzo(k)fluoranthene	µg/Kg		< 100			
Benzo(g,h,i)perylene	µg/Kg		400	48.4 - 620		
Benzo(a)pyrene	µg/Kg		205	0.00 - 353		
Chrysene	µg/Kg		185	13.1 - 339		
Dibenz(a,h)anthracene	µg/Kg		< 100			
Fluoranthene	µg/Kg		876	135 - 1320		

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15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
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ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
PAHs in Soil (Cont.)						
Fluorene	µg/Kg		713	21.0 - 987		
Indeno(1,2,3-cd)pyrene	µg/Kg		< 100			
Naphthalene	µg/Kg		< 100			
Phenanthrene	µg/Kg		< 100			
Pyrene	µg/Kg		< 100			
PCBs in Soil						
Aroclor 1016	mg/Kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1221	mg/Kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1232	mg/Kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1242	mg/Kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1248	mg/Kg	17.3	19.0	4.00 - 29.8	Acceptable	EPA 8082
Aroclor 1254	mg/Kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1260	mg/Kg	< 0.5	0.00		Acceptable	EPA 8082

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ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
CA Volatiles in Soil						
Acetone	µg/Kg		664	0.00 - 2750		
Acetonitrile	µg/Kg		0.00			
Acrolein	µg/Kg		0.00			
Benzene	µg/Kg		3790	2770 - 5490		
Bromobenzene	µg/Kg		0.00			
Bromodichloromethane	µg/Kg		7420	4070 - 11500		
Bromoform	µg/Kg		4420	2320 - 7040		
Bromomethane	µg/Kg		0.00			
2-Butanone (MEK)	µg/Kg		913	0.00 - 2600		
Carbon disulfide	µg/Kg		0.00			
Carbon tetrachloride	µg/Kg		4270	2450 - 6390		
Chloroacetaldehyde	µg/Kg		0.00			
Chlorobenzene	µg/Kg		5160	3940 - 6780		
Chlorodibromomethane	µg/Kg		7030	3990 - 10600		
Chloroethane	µg/Kg		0.00			
bis(2-Chloroethyl)ether	µg/Kg		0.00			
bis(2-Chloroethyloxymethane)	µg/Kg		0.00			
bis(2-Chloroisopropyl)ether	µg/Kg		0.00			
2-Chloroethylvinylether	µg/Kg		0.00			
Chloroform	µg/Kg		6330	4070 - 8810		
Chloromethane	µg/Kg		0.00			
1,2-Dibromo-3-chloropropane	µg/Kg		0.00			
1,2-Dibromoethane (EDB)	µg/Kg		0.00			
Dibromomethane	µg/Kg		0.00			
1,2-Dichlorobenzene	µg/Kg		9900	6010 - 13700		
1,3-Dichlorobenzene	µg/Kg		7060	4310 - 10400		
1,4-Dichlorobenzene	µg/Kg		6190	4310 - 8050		
Dichlorodifluoromethane	µg/Kg		0.00			
1,1-Dichloroethane	µg/Kg		1890	966 - 2930		
1,2-Dichloroethane	µg/Kg		3580	2210 - 5490		
1,1-Dichloroethylene	µg/Kg		0.00			
cis-1,2-Dichloroethylene	µg/Kg		0.00			
trans-1,2-Dichloroethylene	µg/Kg		0.00			
1,2-Dichloropropane	µg/Kg		0.00			
cis-1,3-Dichloropropylene	µg/Kg		0.00			
trans-1,3-Dichloropropylene	µg/Kg		0.00			
Ethylbenzene	µg/Kg		3610	2440 - 5410		
2-Hexanone	µg/Kg		0.00			
Hexylchloroethane	µg/Kg		0.00			
Isopropylbenzene	µg/Kg		0.00			
Methylene chloride	µg/Kg		4570	1800 - 7280		

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ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
CA Volatiles in Soil (Cont.)						
4-Methyl-2-pentanone (MIBK)	µg/Kg		4650	2410 - 7110		
Methyl-t-butylether	µg/Kg		0.00			
Styrene	µg/Kg		0.00			
1,1,1,2-Tetrachloroethane	µg/Kg		2780	1850 - 4170		
1,1,2,2-Tetrachloroethane	µg/Kg		5110	2770 - 7810		
Tetrachloroethylene	µg/Kg		4420	3040 - 6120		
Toluene	µg/Kg		4820	3360 - 6660		
1,2,4-Trichlorobenzene	µg/Kg		0.00			
1,1,1-Trichloroethane	µg/Kg		8520	4500 - 13100		
1,1,2-Trichloroethane	µg/Kg		2550	1730 - 3770		
Trichloroethylene	µg/Kg		3470	2550 - 4860		
Trichlorofluoromethane	µg/Kg		0.00			
1,2,3-Trichloropropane	µg/Kg		3240	830 - 5930		
Vinyl acetate	µg/Kg		0.00			
Vinyl chloride	µg/Kg		0.00			
Total, Xylenes	µg/Kg		8810	6330 - 12600		
Methanol	µg/Kg		0.00			
CA NTK in Soil						
4-amino-2,6-dinitrotoluene	µg/Kg		4180	1530 - 4330		
2-amino-4,6-dinitrotoluene	µg/Kg		0.00			
1-Chloro-2,4-dinitrobenzene	µg/Kg		0.00			
1-Chloro-4-nitrobenzene	µg/Kg		0.00			
4-Chloro-3-nitrotoluene	µg/Kg		0.00			
3,5-Dichloronitrobenzene	µg/Kg		0.00			
1,3-Dinitrobenzene	µg/Kg		8440	4650 - 9810		
1,4-Dinitrobenzene	µg/Kg		0.00			
2,4-Dinitrotoluene	µg/Kg		4810	2740 - 6000		
2,6-Dinitrotoluene	µg/Kg		0.00			
Dinitramine	µg/Kg		0.00			
HMX	µg/Kg		0.00			
1,2-Napthoquinone	µg/Kg		0.00			
1,4-Napthoquinone	µg/Kg		0.00			
Nitrobenzene	µg/Kg		12000	2510 - 16800		
2-Nitrotoluene	µg/Kg		0.00			
3-Nitrotoluene	µg/Kg		0.00			
4-Nitrotoluene	µg/Kg		3900	2220 - 4480		
Pentachloronitrobenzene	µg/Kg		0.00			
RDX	µg/Kg		0.00			
2,3,4,5-Tetrachloronitrobenzene	µg/Kg		0.00			
Tetryl	µg/Kg		0.00			
2,4,6-Trichloronitrobenzene	µg/Kg		0.00			

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ANALYTICAL MANAGEMENT LABS
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EPA ID: KS00902 **Report Issued: 03/28/01**
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ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
CA NTK in Soil (Cont.)						
Trifluralin	µg/Kg		0.00			
1,3,5-Trinitrobenzene	µg/Kg		0.00			
2,4,6-Trinitrotoluene	µg/Kg		8530	4750 - 8590		
CA OHC in Soil						
Diazinon	µg/Kg		0.00			
Dichlorvos (DDVP)	µg/Kg		0.00			
Malathion	µg/Kg		1580	193 - 2710		
Phorate	µg/Kg		2280	0.00 - 3270		
Ronnel	µg/Kg		0.00			
Stirophos (tetrachlorovinphos)	µg/Kg		0.00			
2,4-D	µg/Kg		1200	0.00 - 1400		
2,4-DB	µg/Kg		834	0.00 - 1360		
2,4,5-T	µg/Kg		665	0.00 - 911		
2,4,5-TP (Silvex)	µg/Kg		0.00			
Dalapon	µg/Kg		0.00			
Dicamba	µg/Kg		0.00			
Dinoseb	µg/Kg		0.00			
MCPP	µg/Kg		0.00			
Pentachlorophenol	µg/Kg		0.00			
Aldicarb	µg/Kg		0.00			
Aldicarb sulfone	µg/Kg		0.00			
Aldicarb sulfoxide	µg/Kg		0.00			
Carbaryl	µg/Kg		0.00			
Carbofuran	µg/Kg		733	0.00 - 1080		
Dioxacarb	µg/Kg		0.00			
Diuron	µg/Kg		2160	0.00 - 3270		
3-Hydroxycarbofuran	µg/Kg		0.00			
Methiocarb	µg/Kg		779	0.00 - 2020		
Methomyl	µg/Kg		0.00			
Promecarb	µg/Kg		0.00			
Propoxur	µg/Kg		0.00			
Ignitability						
Ignitability (Flashpoint)	°F		105	93.5 - 133		
Corrosivity (pH)						
Corrosivity (pH)	S.U.		7.79	7.18 - 8.72		
Fluoride in Soil						
Fluoride	mg/Kg		432	46.3 - 432		

Study: **SOIL33**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **ANALYTICAL MANAGEMENT LABS**

Report Type: **Complete**

Report Method: **Method B**

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-33 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil						
Aluminum	mg/Kg		6040	1270 - 10200		
Antimony	mg/Kg		243	0.00 - 243		
Arsenic	mg/Kg		184	103 - 243		
Barium	mg/Kg		148	104 - 198		
Beryllium	mg/Kg		69.5	42.0 - 82.4		
Boron	mg/Kg		133	28.6 - 133		
Cadmium	mg/Kg		219	126 - 248		
Calcium	mg/Kg		3310	1970 - 4430		
Chromium	mg/Kg		117	70.4 - 150		
Cobalt	mg/Kg		70.6	34.5 - 70.6		
Copper	mg/Kg		87.6	60.1 - 113		
Iron	mg/Kg		11800	330 - 21300		
Lead	mg/Kg		110	64.4 - 150		
Magnesium	mg/Kg		1960	881 - 2940		
Manganese	mg/Kg		382	230 - 496		
Mercury	mg/Kg	25.4	28.6	14.1 - 44.7	Acceptable	EPA 7471A
Molybdenum	mg/Kg		92.8	56.0 - 109		
Nickel	mg/Kg		142	85.9 - 168		
Potassium	mg/Kg		1820	621 - 3100		
Selenium	mg/Kg		95.9	51.5 - 124		
Silver	mg/Kg		207	0.00 - 507		
Sodium	mg/Kg		218	0.00 - 573		
Strontium	mg/Kg		182	117 - 225		
Thallium	mg/Kg		166	85.5 - 215		
Tin	mg/Kg		167	55.0 - 167		
Titanium	mg/Kg		282	0.00 - 669		
Vanadium	mg/Kg		84.2	39.9 - 113		
Zinc	mg/Kg		414	261 - 493		
Cyanide in Soil						
Cyanide, total	mg/Kg		277	0.00 - 461		
Reactive Cyanide	mg/Kg		< 25.0			

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ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Hexavalent Chromium in Soil						
Chromium VI	mg/Kg		177	47.5 - 225		

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ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil						
Acenaphthene	µg/Kg		3210	730 - 4210		
Acenaphthylene	µg/Kg		2650	422 - 3040		
Aniline	µg/Kg		0.00			
Anthracene	µg/Kg		0.00			
Benzidine	µg/Kg		0.00			
Benzoic acid	µg/Kg		0.00			
Benzo(a)anthracene	µg/Kg		4170	998 - 5820		
Benzo(b)fluoranthene	µg/Kg		6310	1590 - 8850		
Benzo(k)fluoranthene	µg/Kg		0.00			
Benzo(g,h,i)perylene	µg/Kg		0.00			
Benzo(a)pyrene	µg/Kg		5780	1010 - 6730		
Benzyl alcohol	µg/Kg		0.00			
4-Bromophenyl-phenylether	µg/Kg		6700	1540 - 9400		
Butylbenzylphthalate	µg/Kg		9450	1880 - 13900		
Carbazole	µg/Kg		0.00			
4-Chloroaniline	µg/Kg		0.00			
bis(2-Chloroethoxy)methane	µg/Kg		5820	660 - 7980		
bis(2-Chloroethyl)ether	µg/Kg		0.00			
bis(2-Chloroisopropyl)ether	µg/Kg		1530	0.00 - 2550		
4-Chloro-3-methylphenol	µg/Kg		7390	1110 - 8250		
1-Chloronaphthalene	µg/Kg		0.00			
2-Chloronaphthalene	µg/Kg		3380	745 - 4320		
2-Chlorophenol	µg/Kg		4360	303 - 4740		
4-Chlorophenyl-phenylether	µg/Kg		3710	679 - 5280		
Chrysene	µg/Kg		3700	1020 - 5180		
Dibenz(a,h)anthracene	µg/Kg		0.00			
Dibenzofuran	µg/Kg		2080	472 - 2910		
Di-n-butylphthalate	µg/Kg		4420	1110 - 6710		
1,2-Dichlorobenzene	µg/Kg		0.00			
1,3-Dichlorobenzene	µg/Kg		0.00			
1,4-Dichlorobenzene	µg/Kg		0.00			
3,3'-Dichlorobenzidine	µg/Kg		0.00			
2,4-Dichlorophenol	µg/Kg		4080	553 - 4270		
2,6-Dichlorophenol	µg/Kg		0.00			
Diethylphthalate	µg/Kg		5890	1190 - 8150		
2,4-Dimethylphenol	µg/Kg		6790	0.00 - 6790		
Dimethylphthalate	µg/Kg		0.00			
4,6-Dinitro-2-methylphenol	µg/Kg		0.00			
2,4-Dinitrophenol	µg/Kg		0.00			
2,4-Dinitrotoluene	µg/Kg		6720	1290 - 9030		
2,6-Dinitrotoluene	µg/Kg		7250	2120 - 9320		

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ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)						
Di-n-octylphthalate	µg/Kg		9280	10.0 - 15500		
bis(2-Ethylhexyl)phthalate	µg/Kg		3770	825 - 5780		
Fluoranthene	µg/Kg		5960	1440 - 8400		
Fluorene	µg/Kg		4430	887 - 6170		
Hexachlorobenzene	µg/Kg		2880	780 - 3960		
Hexachlorobutadiene	µg/Kg		2810	168 - 3610		
Hexachlorocyclopentadiene	µg/Kg		5620	0.00 - 5620		
Hexachloroethane	µg/Kg		3600	0.00 - 4340		
Indeno(1,2,3-cd)pyrene	µg/Kg		0.00			
Isophorone	µg/Kg		2660	466 - 3630		
2-Methylnaphthalene	µg/Kg		0.00			
2-Methylphenol	µg/Kg		5040	0.00 - 5040		
3-Methylphenol	µg/Kg		0.00			
4-Methylphenol	µg/Kg		0.00			
Naphthalene	µg/Kg		6010	820 - 7420		
2-Nitroaniline	µg/Kg		0.00			
3-Nitroaniline	µg/Kg		0.00			
4-Nitroaniline	µg/Kg		0.00			
Nitrobenzene	µg/Kg		6000	910 - 7210		
2-Nitrophenol	µg/Kg		0.00			
3-Nitrophenol	µg/Kg		0.00			
4-Nitrophenol	µg/Kg		9820	0.00 - 12000		
N-Nitrosodimethylamine	µg/Kg		0.00			
N-Nitrosodiphenylamine	µg/Kg		0.00			
N-Nitroso-di-n-propylamine	µg/Kg		7290	610 - 9730		
Pentachlorophenol	µg/Kg		0.00			
Phenanthrene	µg/Kg		2450	646 - 3570		
Phenol	µg/Kg		12100	880 - 13500		
Pyrene	µg/Kg		6710	1410 - 9630		
Pyridine	µg/Kg		0.00			
1,2,4-Trichlorobenzene	µg/Kg		3450	275 - 4330		
2,4,5-Trichlorophenol	µg/Kg		3510	414 - 3970		
2,4,6-Trichlorophenol	µg/Kg		3550	559 - 3720		
Additional Base / Neutrals and Acids in Soil						
2-Amino-1-methylbenzene (o-touluidine)	µg/Kg		0.00			
Benzotrichloride	µg/Kg		0.00			
Benzyl chloride	µg/Kg		0.00			
bis(2-methoxyethyl)phthalate	µg/Kg		0.00			
2-Chlorophenyl-4-nitrophenylether	µg/Kg		0.00			
3-Chlorophenyl-4-nitrophenylether	µg/Kg		0.00			
4-Chlorophenyl-4-nitrophenylether	µg/Kg		0.00			

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ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil (Cont.)						
Diamylphthalate	µg/Kg		0.00			
2,4-Dibromophenyl-4-nitrophenylether	µg/Kg		0.00			
2,4-Dichlorophenyl-3-methyl-4-nitrophenylether	µg/Kg		0.00			
2,3-Dichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,4-Dichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,5-Dichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,6-Dichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
3,4-Dichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
3,5-Dichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
Dicyclohexylphthalate	µg/Kg		0.00			
4-Nitrophenylphenylether	µg/Kg		0.00			
Dinonylphthalate	µg/Kg		0.00			
Hexachlorocyclohexane	µg/Kg		0.00			
Hexyl-2-ethylhexylphthalate	µg/Kg		0.00			
Maleic anhydride	µg/Kg		0.00			
Pentachlorobenzene	µg/Kg		0.00			
Pentachlorohexane	µg/Kg		0.00			
1,2,3,4-Tetrachlorobenzene	µg/Kg		0.00			
1,2,3,5-Tetrachlorobenzene	µg/Kg		0.00			
1,2,4,5-Tetrachlorobenzene	µg/Kg		0.00			
2,3,4,5-Tetrachlorophenol	µg/Kg		0.00			
2,3,4,6-Tetrachlorophenol	µg/Kg		0.00			
2,3,5,6-Tetrachlorophenol	µg/Kg		0.00			
1,2,3-Trichlorobenzene	µg/Kg		0.00			
1,3,5-Trichlorobenzene	µg/Kg		0.00			
2,3,4-Trichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,3,5-Trichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,3,6-Trichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,4,5-Trichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,4,6-Trichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
3,4,5-Trichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
Pesticides in Soil						
Aldrin	µg/Kg	132	151	69.5 - 205	Acceptable	EPA 8081A
alpha-BHC	µg/Kg	111	128	55.2 - 173	Acceptable	EPA 8081A
beta-BHC	µg/Kg	197	265	95.9 - 346	Acceptable	EPA 8081A
delta-BHC	µg/Kg	243	341	143 - 453	Acceptable	EPA 8081A
gamma-BHC(Lindane)	µg/Kg	90.7	107	46.9 - 146	Acceptable	EPA 8081A
alpha-Chlordane	µg/Kg	104	148	22.2 - 236	Acceptable	EPA 8081A
gamma-Chlordane	µg/Kg	1.42	0.00		Not Acceptable	EPA 8081A
4,4'-DDD	µg/Kg	78.7	96.2	32.5 - 160	Acceptable	EPA 8081A
4,4'-DDE	µg/Kg	214	232	83.5 - 339	Acceptable	EPA 8081A

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ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Pesticides in Soil (Cont.)						
4,4'-DDT	µg/Kg	213	193	63.3 - 303	Acceptable	EPA 8081A
Dieldrin	µg/Kg	252	319	140 - 446	Acceptable	EPA 8081A
Endrin	µg/Kg	135	140	58.5 - 218	Acceptable	EPA 8081A
Endrin aldehyde	µg/Kg	8.91	0.00		Not Acceptable	EPA 8081A
Endrin ketone	µg/Kg	5.2	0.00		Not Acceptable	EPA 8081A
Endosulfan I	µg/Kg	226	317	125 - 413	Acceptable	EPA 8081A
Endosulfan II	µg/Kg	77.9	217	67.5 - 233	Acceptable	EPA 8081A
Endosulfan sulfate	µg/Kg	116	279	85.4 - 417	Acceptable	EPA 8081A
Heptachlor	µg/Kg	239	321	116 - 438	Acceptable	EPA 8081A
Heptachlor epoxide	µg/Kg	106	110	49.4 - 171	Acceptable	EPA 8081A
Methoxychlor	µg/Kg	199	412	13.0 - 721	Acceptable	EPA 8081A
Chlordane in Soil						
Chlordane, Technical	µg/Kg		429	61.0 - 631		
Toxaphene in Soil						
Toxaphene	µg/Kg		215	0.00 - 419		
Volatiles in Soil						
Acetone	µg/Kg		61.3	0.00 - 324		
Acetonitrile	µg/Kg		0.00			
Acrolein	µg/Kg		0.00			
Benzene	µg/Kg		75.8	45.9 - 117		
Bromodichloromethane	µg/Kg		148	75.0 - 231		
Bromoform	µg/Kg		88.5	41.4 - 147		
Bromomethane	µg/Kg		0.00			
2-Butanone (MEK)	µg/Kg		19.7	0.00 - 75.8		
Carbon disulfide	µg/Kg		0.00			
Carbon tetrachloride	µg/Kg		85.4	35.5 - 141		
Chlorobenzene	µg/Kg		103	49.2 - 155		
Chlorodibromomethane	µg/Kg		141	74.9 - 217		
Chloroethane	µg/Kg		0.00			
2-Chloroethylvinylether	µg/Kg		0.00			
Chloroform	µg/Kg		127	70.8 - 181		
Chloromethane	µg/Kg		0.00			
1,2-Dibromo-3-chloropropane	µg/Kg		0.00			
1,2-Dibromoethane (EDB)	µg/Kg		0.00			
Dibromomethane	µg/Kg		0.00			
1,2-Dichlorobenzene	µg/Kg		198	53.4 - 313		
1,3-Dichlorobenzene	µg/Kg		141	46.1 - 216		
1,4-Dichlorobenzene	µg/Kg		124	33.4 - 191		
Dichlorodifluoromethane	µg/Kg		0.00			
1,1-Dichloroethane	µg/Kg		37.9	21.4 - 55.9		
1,2-Dichloroethane	µg/Kg		71.5	42.6 - 109		

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ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)						
1,1-Dichloroethylene	µg/Kg		0.00			
cis-1,2-Dichloroethylene	µg/Kg		0.00			
trans-1,2-Dichloroethylene	µg/Kg		0.00			
1,2-Dichloropropane	µg/Kg		0.00			
cis-1,3-Dichloropropylene	µg/Kg		0.00			
trans-1,3-Dichloropropylene	µg/Kg		0.00			
Ethylbenzene	µg/Kg		72.2	36.0 - 116		
2-Hexanone	µg/Kg		0.00			
Methylene chloride	µg/Kg		91.3	40.3 - 147		
4-Methyl-2-pentanone (MIBK)	µg/Kg		97.4	31.9 - 166		
Styrene	µg/Kg		0.00			
1,1,1,2-Tetrachloroethane	µg/Kg		55.6	29.0 - 89.0		
1,1,2,2-Tetrachloroethane	µg/Kg		102	45.9 - 158		
Tetrachloroethylene	µg/Kg		88.5	37.4 - 130		
Toluene	µg/Kg		96.4	48.4 - 150		
1,1,1-Trichloroethane	µg/Kg		170	99.0 - 243		
1,1,2-Trichloroethane	µg/Kg		50.9	33.0 - 76.0		
Trichloroethylene	µg/Kg		69.3	34.5 - 110		
Trichlorofluoromethane	µg/Kg		0.00			
1,2,3-Trichloropropane	µg/Kg		64.9	20.0 - 109		
Vinyl acetate	µg/Kg		0.00			
Vinyl chloride	µg/Kg		0.00			
Total, Xylenes	µg/Kg		176	65.8 - 290		
Herbicides in Soil						
2,4-D	µg/Kg		390	0.00 - 822		
Dicamba	µg/Kg		212	0.00 - 392		
Disulfoton	µg/Kg		434	0.00 - 683		
2,4,5-T	µg/Kg		296	0.00 - 686		
2,4,5-TP (Silvex)	µg/Kg		139	0.00 - 284		
PAHs in Soil						
Acenaphthene	µg/Kg		< 100			
Acenaphthylene	µg/Kg		< 100			
Anthracene	µg/Kg		3280	0.00 - 4260		
Benzo(a)anthracene	µg/Kg		441	0.00 - 744		
Benzo(b)fluoranthene	µg/Kg		265	0.00 - 537		
Benzo(k)fluoranthene	µg/Kg		< 100			
Benzo(g,h,i)perylene	µg/Kg		400	48.4 - 620		
Benzo(a)pyrene	µg/Kg		205	0.00 - 353		
Chrysene	µg/Kg		185	13.1 - 339		
Dibenz(a,h)anthracene	µg/Kg		< 100			
Fluoranthene	µg/Kg		876	135 - 1320		

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PAHs in Soil (Cont.)						
Fluorene	µg/Kg		713	21.0 - 987		
Indeno(1,2,3-cd)pyrene	µg/Kg		< 100			
Naphthalene	µg/Kg		< 100			
Phenanthrene	µg/Kg		< 100			
Pyrene	µg/Kg		< 100			
PCBs in Soil						
Aroclor 1016	mg/Kg		0.00			
Aroclor 1221	mg/Kg		0.00			
Aroclor 1232	mg/Kg		0.00			
Aroclor 1242	mg/Kg		0.00			
Aroclor 1248	mg/Kg		19.0	4.00 - 29.8		
Aroclor 1254	mg/Kg		0.00			
Aroclor 1260	mg/Kg		0.00			

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ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
CA Volatiles in Soil						
Acetone	µg/Kg		664	0.00 - 2750		
Acetonitrile	µg/Kg		0.00			
Acrolein	µg/Kg		0.00			
Benzene	µg/Kg		3790	2770 - 5490		
Bromobenzene	µg/Kg		0.00			
Bromodichloromethane	µg/Kg		7420	4070 - 11500		
Bromoform	µg/Kg		4420	2320 - 7040		
Bromomethane	µg/Kg		0.00			
2-Butanone (MEK)	µg/Kg		913	0.00 - 2600		
Carbon disulfide	µg/Kg		0.00			
Carbon tetrachloride	µg/Kg		4270	2450 - 6390		
Chloroacetaldehyde	µg/Kg		0.00			
Chlorobenzene	µg/Kg		5160	3940 - 6780		
Chlorodibromomethane	µg/Kg		7030	3990 - 10600		
Chloroethane	µg/Kg		0.00			
bis(2-Chloroethyl)ether	µg/Kg		0.00			
bis(2-Chloroethyloxymethane)	µg/Kg		0.00			
bis(2-Chloroisopropyl)ether	µg/Kg		0.00			
2-Chloroethylvinylether	µg/Kg		0.00			
Chloroform	µg/Kg		6330	4070 - 8810		
Chloromethane	µg/Kg		0.00			
1,2-Dibromo-3-chloropropane	µg/Kg		0.00			
1,2-Dibromoethane (EDB)	µg/Kg		0.00			
Dibromomethane	µg/Kg		0.00			
1,2-Dichlorobenzene	µg/Kg		9900	6010 - 13700		
1,3-Dichlorobenzene	µg/Kg		7060	4310 - 10400		
1,4-Dichlorobenzene	µg/Kg		6190	4310 - 8050		
Dichlorodifluoromethane	µg/Kg		0.00			
1,1-Dichloroethane	µg/Kg		1890	966 - 2930		
1,2-Dichloroethane	µg/Kg		3580	2210 - 5490		
1,1-Dichloroethylene	µg/Kg		0.00			
cis-1,2-Dichloroethylene	µg/Kg		0.00			
trans-1,2-Dichloroethylene	µg/Kg		0.00			
1,2-Dichloropropane	µg/Kg		0.00			
cis-1,3-Dichloropropylene	µg/Kg		0.00			
trans-1,3-Dichloropropylene	µg/Kg		0.00			
Ethylbenzene	µg/Kg		3610	2440 - 5410		
2-Hexanone	µg/Kg		0.00			
Hexylchloroethane	µg/Kg		0.00			
Isopropylbenzene	µg/Kg		0.00			
Methylene chloride	µg/Kg		4570	1800 - 7280		

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ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
CA Volatiles in Soil (Cont.)						
4-Methyl-2-pentanone (MIBK)	µg/Kg		4650	2410 - 7110		
Methyl-t-butylether	µg/Kg		0.00			
Styrene	µg/Kg		0.00			
1,1,1,2-Tetrachloroethane	µg/Kg		2780	1850 - 4170		
1,1,2,2-Tetrachloroethane	µg/Kg		5110	2770 - 7810		
Tetrachloroethylene	µg/Kg		4420	3040 - 6120		
Toluene	µg/Kg		4820	3360 - 6660		
1,2,4-Trichlorobenzene	µg/Kg		0.00			
1,1,1-Trichloroethane	µg/Kg		8520	4500 - 13100		
1,1,2-Trichloroethane	µg/Kg		2550	1730 - 3770		
Trichloroethylene	µg/Kg		3470	2550 - 4860		
Trichlorofluoromethane	µg/Kg		0.00			
1,2,3-Trichloropropane	µg/Kg		3240	830 - 5930		
Vinyl acetate	µg/Kg		0.00			
Vinyl chloride	µg/Kg		0.00			
Total, Xylenes	µg/Kg		8810	6330 - 12600		
Methanol	µg/Kg		0.00			
CA NTK in Soil						
4-amino-2,6-dinitrotoluene	µg/Kg		4180	1530 - 4330		
2-amino-4,6-dinitrotoluene	µg/Kg		0.00			
1-Chloro-2,4-dinitrobenzene	µg/Kg		0.00			
1-Chloro-4-nitrobenzene	µg/Kg		0.00			
4-Chloro-3-nitrotoluene	µg/Kg		0.00			
3,5-Dichloronitrobenzene	µg/Kg		0.00			
1,3-Dinitrobenzene	µg/Kg		8440	4650 - 9810		
1,4-Dinitrobenzene	µg/Kg		0.00			
2,4-Dinitrotoluene	µg/Kg		4810	2740 - 6000		
2,6-Dinitrotoluene	µg/Kg		0.00			
Dinitramine	µg/Kg		0.00			
HMX	µg/Kg		0.00			
1,2-Napthoquinone	µg/Kg		0.00			
1,4-Napthoquinone	µg/Kg		0.00			
Nitrobenzene	µg/Kg		12000	2510 - 16800		
2-Nitrotoluene	µg/Kg		0.00			
3-Nitrotoluene	µg/Kg		0.00			
4-Nitrotoluene	µg/Kg		3900	2220 - 4480		
Pentachloronitrobenzene	µg/Kg		0.00			
RDX	µg/Kg		0.00			
2,3,4,5-Tetrachloronitrobenzene	µg/Kg		0.00			
Tetryl	µg/Kg		0.00			
2,4,6-Trichloronitrobenzene	µg/Kg		0.00			

NVLAP® Lab code: 200388-0

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
CA NTK in Soil (Cont.)						
Trifluralin	µg/Kg		0.00			
1,3,5-Trinitrobenzene	µg/Kg		0.00			
2,4,6-Trinitrotoluene	µg/Kg		8530	4750 - 8590		
CA OHC in Soil						
Diazinon	µg/Kg		0.00			
Dichlorvos (DDVP)	µg/Kg		0.00			
Malathion	µg/Kg		1580	193 - 2710		
Phorate	µg/Kg		2280	0.00 - 3270		
Ronnel	µg/Kg		0.00			
Stirophos (tetrachlorovinphos)	µg/Kg		0.00			
2,4-D	µg/Kg		1200	0.00 - 1400		
2,4-DB	µg/Kg		834	0.00 - 1360		
2,4,5-T	µg/Kg		665	0.00 - 911		
2,4,5-TP (Silvex)	µg/Kg		0.00			
Dalapon	µg/Kg		0.00			
Dicamba	µg/Kg		0.00			
Dinoseb	µg/Kg		0.00			
MCPP	µg/Kg		0.00			
Pentachlorophenol	µg/Kg		0.00			
Aldicarb	µg/Kg		0.00			
Aldicarb sulfone	µg/Kg		0.00			
Aldicarb sulfoxide	µg/Kg		0.00			
Carbaryl	µg/Kg		0.00			
Carbofuran	µg/Kg		733	0.00 - 1080		
Dioxacarb	µg/Kg		0.00			
Diuron	µg/Kg		2160	0.00 - 3270		
3-Hydroxycarbofuran	µg/Kg		0.00			
Methiocarb	µg/Kg		779	0.00 - 2020		
Methomyl	µg/Kg		0.00			
Promecarb	µg/Kg		0.00			
Propoxur	µg/Kg		0.00			
Ignitability						
Ignitability (Flashpoint)	°F		105	93.5 - 133		
Corrosivity (pH)						
Corrosivity (pH)	S.U.		7.79	7.18 - 8.72		
Fluoride in Soil						
Fluoride	mg/Kg		432	46.3 - 432		

Study: **SOIL33**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **ANALYTICAL MANAGEMENT LABS**

Report Type: **NELAC**

Report Method: **Method A**

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-33 Final NELAC Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil						
Aluminum	mg/Kg	5852	6040	1270 - 10200	Acceptable	EPA 6010B
Antimony	mg/Kg	210	243	0.00 - 243	Acceptable	EPA 6010B
Arsenic	mg/Kg	188	184	103 - 243	Acceptable	EPA 6010B
Barium	mg/Kg	155	148	104 - 198	Acceptable	EPA 6010B
Beryllium	mg/Kg	61.7	69.5	42.0 - 82.4	Acceptable	EPA 6010B
Boron	mg/Kg		133	28.6 - 133		
Cadmium	mg/Kg	198	219	126 - 248	Acceptable	EPA 6010B
Calcium	mg/Kg	3382	3310	1970 - 4430	Acceptable	EPA 6010B
Chromium	mg/Kg	119	117	70.4 - 150	Acceptable	EPA 6010B
Cobalt	mg/Kg	51.5	70.6	34.5 - 70.6	Acceptable	EPA 6010B
Copper	mg/Kg	87.1	87.6	60.1 - 113	Acceptable	EPA 6010B
Iron	mg/Kg	10203	11800	333 - 21300	Acceptable	EPA 6010B
Lead	mg/Kg	104	110	64.4 - 150	Acceptable	EPA 6010B
Magnesium	mg/Kg	1908	1960	881 - 2940	Acceptable	EPA 6010B
Manganese	mg/Kg	370	382	230 - 496	Acceptable	EPA 6010B
Mercury	mg/Kg		28.6	14.1 - 44.7		
Molybdenum	mg/Kg	86.7	92.8	56.0 - 109	Acceptable	EPA 6010B
Nickel	mg/Kg	139	142	85.9 - 168	Acceptable	EPA 6010B
Potassium	mg/Kg	1886	1820	621 - 3100	Acceptable	EPA 6010B
Selenium	mg/Kg	91.5	95.9	51.5 - 124	Acceptable	EPA 6010B
Silver	mg/Kg	72.9	207	0.00 - 507	Acceptable	EPA 6010B
Sodium	mg/Kg	223	218	0.00 - 573	Acceptable	EPA 6010B
Thallium	mg/Kg	158	166	85.5 - 215	Acceptable	EPA 6010B
Tin	mg/Kg		167	55.0 - 167		
Vanadium	mg/Kg	76.2	84.2	39.9 - 113	Acceptable	EPA 6010B
Zinc	mg/Kg	396	414	261 - 493	Acceptable	EPA 6010B
Cyanide in Soil						
Cyanide, total	mg/Kg	136	277	0.00 - 461	Acceptable	EPA 335.2

NVLAP®

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Hexavalent Chromium in Soil						
Chromium VI	mg/Kg	96.9	177	47.5 - 225	Acceptable	EPA 3060A

NVLAP® Lab code: 200386-0

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil						
Acenaphthene	µg/Kg	3037	3210	730 - 4210	Acceptable	EPA 8270C
Acenaphthylene	µg/Kg	2138	2650	422 - 3040	Acceptable	EPA 8270C
Anthracene	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
Benzo(a)anthracene	µg/Kg	3330	4170	998 - 5820	Acceptable	EPA 8270C
Benzo(b)fluoranthene	µg/Kg	5293	6310	1590 - 8850	Acceptable	EPA 8270C
Benzo(k)fluoranthene	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
Benzo(g,h,i)perylene	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
Benzo(a)pyrene	µg/Kg	3678	5780	1010 - 6730	Acceptable	EPA 8270C
4-Bromophenyl-phenylether	µg/Kg	5309	6700	1540 - 9400	Acceptable	EPA 8270C
Butylbenzylphthalate	µg/Kg	8150	9450	1880 - 13900	Acceptable	EPA 8270C
bis(2-Chloroethoxy)methane	µg/Kg	5101	5820	660 - 7980	Acceptable	EPA 8270C
bis(2-Chloroisopropyl)ether	µg/Kg	< 66.7	1530	0.00 - 2550	Acceptable	EPA 8270C
4-Chloro-3-methylphenol	µg/Kg	4745	7390	1110 - 8250	Acceptable	EPA 8270C
2-Chloronaphthalene	µg/Kg	2292	3380	745 - 4320	Acceptable	EPA 8270C
2-Chlorophenol	µg/Kg	2786	4360	303 - 4740	Acceptable	EPA 8270C
4-Chlorophenyl-phenylether	µg/Kg	3498	3710	679 - 5280	Acceptable	EPA 8270C
Chrysene	µg/Kg	3023	3700	1020 - 5180	Acceptable	EPA 8270C
Dibenz(a,h)anthracene	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
Dibenzofuran	µg/Kg	1795	2080	472 - 2910	Acceptable	EPA 8270C
Di-n-butylphthalate	µg/Kg	4874	4420	1110 - 6710	Acceptable	EPA 8270C
2,4-Dichlorophenol	µg/Kg	2365	4080	553 - 4270	Acceptable	EPA 8270C
Diethylphthalate	µg/Kg	4623	5890	1190 - 8150	Acceptable	EPA 8270C
2,4-Dimethylphenol	µg/Kg	< 66.7	6790	0.00 - 6790	Acceptable	EPA 8270C
Dimethylphthalate	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
4,6-Dinitro-2-methylphenol	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
2,4-Dinitrophenol	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
2,4-Dinitrotoluene	µg/Kg	5474	6720	1290 - 9030	Acceptable	EPA 8270C
2,6-Dinitrotoluene	µg/Kg	5864	7250	2120 - 9320	Acceptable	EPA 8270C
Di-n-octylphthalate	µg/Kg	7438	9280	10.0 - 15500	Acceptable	EPA 8270C
bis(2-Ethylhexyl)phthalate	µg/Kg	3619	3770	825 - 5780	Acceptable	EPA 8270C
Fluoranthene	µg/Kg	5528	5960	1440 - 8400	Acceptable	EPA 8270C
Fluorene	µg/Kg	4145	4430	887 - 6170	Acceptable	EPA 8270C
Hexachlorobenzene	µg/Kg	2436	2880	780 - 3960	Acceptable	EPA 8270C
Hexachlorobutadiene	µg/Kg	2263	2810	168 - 3610	Acceptable	EPA 8270C
Hexachlorocyclopentadiene	µg/Kg	72.7	5620	0.00 - 5620	Acceptable	EPA 8270C
Hexachloroethane	µg/Kg	2470	3600	0.00 - 4340	Acceptable	EPA 8270C
Indeno(1,2,3-cd)pyrene	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
Isophorone	µg/Kg	2547	2660	466 - 3630	Acceptable	EPA 8270C
2-Methylphenol	µg/Kg	1519	5040	0.00 - 5040	Acceptable	EPA 8270C
Naphthalene	µg/Kg	5168	6010	820 - 7420	Acceptable	EPA 8270C
Nitrobenzene	µg/Kg	4271	6000	910 - 7210	Acceptable	EPA 8270C

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)						
2-Nitrophenol	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
4-Nitrophenol	µg/Kg	4983	9820	0.00 - 12000	Acceptable	EPA 8270C
N-Nitroso-di-n-propylamine	µg/Kg	14497	7290	610 - 9730	Not Acceptable	EPA 8270C
Pentachlorophenol	µg/Kg	< 66.7	0.00		Acceptable	EPA 8270C
Phenanthrene	µg/Kg	2365	2450	646 - 3570	Acceptable	EPA 8270C
Phenol	µg/Kg	8878	12100	880 - 13500	Acceptable	EPA 8270C
Pyrene	µg/Kg	5528	6710	1410 - 9630	Acceptable	EPA 8270C
1,2,4-Trichlorobenzene	µg/Kg		3450	275 - 4330		
2,4,5-Trichlorophenol	µg/Kg	2105	3510	414 - 3970	Acceptable	EPA 8270C
2,4,6-Trichlorophenol	µg/Kg	2100	3550	559 - 3720	Acceptable	EPA 8270C
Pesticides in Soil						
Aldrin	µg/Kg		151	69.5 - 205		
alpha-BHC	µg/Kg		128	55.2 - 173		
beta-BHC	µg/Kg		265	95.9 - 346		
delta-BHC	µg/Kg		341	143 - 453		
gamma-BHC(Lindane)	µg/Kg		107	46.9 - 146		
4,4'-DDD	µg/Kg		96.2	32.5 - 160		
4,4'-DDE	µg/Kg		232	83.5 - 339		
4,4'-DDT	µg/Kg		193	63.3 - 303		
Dieldrin	µg/Kg		319	140 - 446		
Endrin	µg/Kg		140	58.5 - 218		
Endrin aldehyde	µg/Kg		0.00			
Endosulfan I	µg/Kg		317	125 - 413		
Endosulfan II	µg/Kg		217	67.5 - 233		
Endosulfan sulfate	µg/Kg		279	85.4 - 417		
Heptachlor	µg/Kg		321	116 - 438		
Heptachlor epoxide	µg/Kg		110	49.4 - 171		
Methoxychlor	µg/Kg		412	13.0 - 721		
Chlordane in Soil						
Chlordane, Technical	µg/Kg	554	429	61.0 - 631	Acceptable	EPA 8081A
Toxaphene in Soil						
Toxaphene	µg/Kg	261	215	0.00 - 419	Acceptable	EPA 8081A
Volatiles in Soil						
Benzene	µg/Kg	75.2	75.8	45.9 - 117	Acceptable	EPA 8260B
Bromodichloromethane	µg/Kg	135	148	75.0 - 231	Acceptable	EPA 8260B
Bromoform	µg/Kg	89.4	88.5	41.4 - 147	Acceptable	EPA 8260B
Carbon tetrachloride	µg/Kg	77.9	85.4	35.5 - 141	Acceptable	EPA 8260B
Chlorobenzene	µg/Kg	92.3	103	49.2 - 155	Acceptable	EPA 8260B
Chlorodibromomethane	µg/Kg	137	141	74.9 - 217	Acceptable	EPA 8260B
Chloroform	µg/Kg	113	127	70.8 - 181	Acceptable	EPA 8260B
1,2-Dichlorobenzene	µg/Kg	154	198	53.4 - 313	Acceptable	EPA 8260B

NVLAP® Lab code: 200386-0

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)						
1,3-Dichlorobenzene	µg/Kg	108	141	46.1 - 216	Acceptable	EPA 8260B
1,4-Dichlorobenzene	µg/Kg	95	124	33.4 - 191	Acceptable	EPA 8260B
1,1-Dichloroethane	µg/Kg	31.7	37.9	21.4 - 55.9	Acceptable	EPA 8260B
1,2-Dichloroethane	µg/Kg	64.9	71.5	42.6 - 109	Acceptable	EPA 8260B
Ethylbenzene	µg/Kg	68.6	72.2	36.0 - 116	Acceptable	EPA 8260B
Methylene chloride	µg/Kg	81.0	91.3	40.3 - 147	Acceptable	EPA 8260B
4-Methyl-2-pentanone (MIBK)	µg/Kg	111	97.4	31.9 - 166	Acceptable	EPA 8260B
1,1,1,2-Tetrachloroethane	µg/Kg	51.7	55.6	29.0 - 89.0	Acceptable	EPA 8260B
1,1,2,2-Tetrachloroethane	µg/Kg	94.8	102	45.9 - 158	Acceptable	EPA 8260B
Tetrachloroethylene	µg/Kg	83.9	88.5	37.4 - 130	Acceptable	EPA 8260B
Toluene	µg/Kg	93.3	96.4	48.4 - 150	Acceptable	EPA 8260B
1,1,1-Trichloroethane	µg/Kg	160	170	99.0 - 243	Acceptable	EPA 8260B
Trichloroethylene	µg/Kg	75.2	69.3	34.5 - 110	Acceptable	EPA 8260B
Total, Xylenes	µg/Kg	164	176	65.8 - 290	Acceptable	EPA 8260B
Herbicides in Soil						
2,4-D	µg/Kg		390	0.00 - 822		
Dicamba	µg/Kg		212	0.00 - 392		
Disulfoton	µg/Kg		434	0.00 - 683		
2,4,5-T	µg/Kg		296	0.00 - 686		
2,4,5-TP (Silvex)	µg/Kg		139	0.00 - 284		
PCBs in Soil						
Aroclor 1016	mg/Kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1221	mg/Kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1232	mg/Kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1242	mg/Kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1248	mg/Kg	17.3	19.0	4.00 - 29.8	Acceptable	EPA 8082
Aroclor 1254	mg/Kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1260	mg/Kg	< 0.5	0.00		Acceptable	EPA 8082

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
CA Volatiles in Soil						
Benzene	µg/Kg		3790	2770 - 5490		
Bromodichloromethane	µg/Kg		7420	4070 - 11500		
Bromoform	µg/Kg		4420	2320 - 7040		
Carbon tetrachloride	µg/Kg		4270	2450 - 6390		
Chlorobenzene	µg/Kg		5160	3940 - 6780		
Chlorodibromomethane	µg/Kg		7030	3990 - 10600		
Chloroform	µg/Kg		6330	4070 - 8810		
1,2-Dichlorobenzene	µg/Kg		9900	6010 - 13700		
1,3-Dichlorobenzene	µg/Kg		7060	4310 - 10400		
1,4-Dichlorobenzene	µg/Kg		6190	4310 - 8050		
1,1-Dichloroethane	µg/Kg		1890	966 - 2930		
1,2-Dichloroethane	µg/Kg		3580	2210 - 5490		
Ethylbenzene	µg/Kg		3610	2440 - 5410		
Methylene chloride	µg/Kg		4570	1800 - 7280		
4-Methyl-2-pentanone (MIBK)	µg/Kg		4650	2410 - 7110		
1,1,1,2-Tetrachloroethane	µg/Kg		2780	1850 - 4170		
1,1,2,2-Tetrachloroethane	µg/Kg		5110	2770 - 7810		
Tetrachloroethylene	µg/Kg		4420	3040 - 6120		
Toluene	µg/Kg		4820	3360 - 6660		
1,1,1-Trichloroethane	µg/Kg		8520	4500 - 13100		
Trichloroethylene	µg/Kg		3470	2550 - 4860		
Total, Xylenes	µg/Kg		8810	6330 - 12600		

NVLAP®
Lab code: 200086-0

Study: **SOIL33**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **ANALYTICAL MANAGEMENT LABS**

Report Type: **NELAC**

Report Method: **Method B**

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-33 Final NELAC Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil						
Aluminum	mg/Kg		6040	1270 - 10200		
Antimony	mg/Kg		243	0.00 - 243		
Arsenic	mg/Kg		184	103 - 243		
Barium	mg/Kg		148	104 - 198		
Beryllium	mg/Kg		69.5	42.0 - 82.4		
Boron	mg/Kg		133	28.6 - 133		
Cadmium	mg/Kg		219	126 - 248		
Calcium	mg/Kg		3310	1970 - 4430		
Chromium	mg/Kg		117	70.4 - 150		
Cobalt	mg/Kg		70.6	34.5 - 70.6		
Copper	mg/Kg		87.6	60.1 - 113		
Iron	mg/Kg		11800	333 - 21300		
Lead	mg/Kg		110	64.4 - 150		
Magnesium	mg/Kg		1960	881 - 2940		
Manganese	mg/Kg		382	230 - 496		
Mercury	mg/Kg	25.4	28.6	14.1 - 44.7	Acceptable	EPA 7471A
Molybdenum	mg/Kg		92.8	56.0 - 109		
Nickel	mg/Kg		142	85.9 - 168		
Potassium	mg/Kg		1820	621 - 3100		
Selenium	mg/Kg		95.9	51.5 - 124		
Silver	mg/Kg		207	0.00 - 507		
Sodium	mg/Kg		218	0.00 - 573		
Thallium	mg/Kg		166	85.5 - 215		
Tin	mg/Kg		167	55.0 - 167		
Vanadium	mg/Kg		84.2	39.9 - 113		
Zinc	mg/Kg		414	261 - 493		
Cyanide in Soil						
Cyanide, total	mg/Kg		277	0.00 - 461		

NVLAP®

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Hexavalent Chromium in Soil						
Chromium VI	mg/Kg		177	47.5 - 225		

NVLAP® Lab code: 200386-0

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil						
Acenaphthene	µg/Kg		3210	730 - 4210		
Acenaphthylene	µg/Kg		2650	422 - 3040		
Anthracene	µg/Kg		0.00			
Benzo(a)anthracene	µg/Kg		4170	998 - 5820		
Benzo(b)fluoranthene	µg/Kg		6310	1590 - 8850		
Benzo(k)fluoranthene	µg/Kg		0.00			
Benzo(g,h,i)perylene	µg/Kg		0.00			
Benzo(a)pyrene	µg/Kg		5780	1010 - 6730		
4-Bromophenyl-phenylether	µg/Kg		6700	1540 - 9400		
Butylbenzylphthalate	µg/Kg		9450	1880 - 13900		
bis(2-Chloroethoxy)methane	µg/Kg		5820	660 - 7980		
bis(2-Chloroisopropyl)ether	µg/Kg		1530	0.00 - 2550		
4-Chloro-3-methylphenol	µg/Kg		7390	1110 - 8250		
2-Chloronaphthalene	µg/Kg		3380	745 - 4320		
2-Chlorophenol	µg/Kg		4360	303 - 4740		
4-Chlorophenyl-phenylether	µg/Kg		3710	679 - 5280		
Chrysene	µg/Kg		3700	1020 - 5180		
Dibenz(a,h)anthracene	µg/Kg		0.00			
Dibenzofuran	µg/Kg		2080	472 - 2910		
Di-n-butylphthalate	µg/Kg		4420	1110 - 6710		
2,4-Dichlorophenol	µg/Kg		4080	553 - 4270		
Diethylphthalate	µg/Kg		5890	1190 - 8150		
2,4-Dimethylphenol	µg/Kg		6790	0.00 - 6790		
Dimethylphthalate	µg/Kg		0.00			
4,6-Dinitro-2-methylphenol	µg/Kg		0.00			
2,4-Dinitrophenol	µg/Kg		0.00			
2,4-Dinitrotoluene	µg/Kg		6720	1290 - 9030		
2,6-Dinitrotoluene	µg/Kg		7250	2120 - 9320		
Di-n-octylphthalate	µg/Kg		9280	10.0 - 15500		
bis(2-Ethylhexyl)phthalate	µg/Kg		3770	825 - 5780		
Fluoranthene	µg/Kg		5960	1440 - 8400		
Fluorene	µg/Kg		4430	887 - 6170		
Hexachlorobenzene	µg/Kg		2880	780 - 3960		
Hexachlorobutadiene	µg/Kg		2810	168 - 3610		
Hexachlorocyclopentadiene	µg/Kg		5620	0.00 - 5620		
Hexachloroethane	µg/Kg		3600	0.00 - 4340		
Indeno(1,2,3-cd)pyrene	µg/Kg		0.00			
Isophorone	µg/Kg		2660	466 - 3630		
2-Methylphenol	µg/Kg		5040	0.00 - 5040		
Naphthalene	µg/Kg		6010	820 - 7420		
Nitrobenzene	µg/Kg		6000	910 - 7210		

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)						
2-Nitrophenol	µg/Kg		0.00			
4-Nitrophenol	µg/Kg		9820	0.00 - 12000		
N-Nitroso-di-n-propylamine	µg/Kg		7290	610 - 9730		
Pentachlorophenol	µg/Kg		0.00			
Phenanthrene	µg/Kg		2450	646 - 3570		
Phenol	µg/Kg		12100	880 - 13500		
Pyrene	µg/Kg		6710	1410 - 9630		
1,2,4-Trichlorobenzene	µg/Kg		3450	275 - 4330		
2,4,5-Trichlorophenol	µg/Kg		3510	414 - 3970		
2,4,6-Trichlorophenol	µg/Kg		3550	559 - 3720		
Pesticides in Soil						
Aldrin	µg/Kg	132	151	69.5 - 205	Acceptable	EPA 8081A
alpha-BHC	µg/Kg	111	128	55.2 - 173	Acceptable	EPA 8081A
beta-BHC	µg/Kg	197	265	95.9 - 346	Acceptable	EPA 8081A
delta-BHC	µg/Kg	243	341	143 - 453	Acceptable	EPA 8081A
gamma-BHC(Lindane)	µg/Kg	90.7	107	46.9 - 146	Acceptable	EPA 8081A
4,4'-DDD	µg/Kg	78.7	96.2	32.5 - 160	Acceptable	EPA 8081A
4,4'-DDE	µg/Kg	214	232	83.5 - 339	Acceptable	EPA 8081A
4,4'-DDT	µg/Kg	213	193	63.3 - 303	Acceptable	EPA 8081A
Dieldrin	µg/Kg	252	319	140 - 446	Acceptable	EPA 8081A
Endrin	µg/Kg	135	140	58.5 - 218	Acceptable	EPA 8081A
Endrin aldehyde	µg/Kg	8.91	0.00		Not Acceptable	EPA 8081A
Endosulfan I	µg/Kg	226	317	125 - 413	Acceptable	EPA 8081A
Endosulfan II	µg/Kg	77.9	217	67.5 - 233	Acceptable	EPA 8081A
Endosulfan sulfate	µg/Kg	116	279	85.4 - 417	Acceptable	EPA 8081A
Heptachlor	µg/Kg	239	321	116 - 438	Acceptable	EPA 8081A
Heptachlor epoxide	µg/Kg	106	110	49.4 - 171	Acceptable	EPA 8081A
Methoxychlor	µg/Kg	199	412	13.0 - 721	Acceptable	EPA 8081A
Chlordane in Soil						
Chlordane, Technical	µg/Kg		429	61.0 - 631		
Toxaphene in Soil						
Toxaphene	µg/Kg		215	0.00 - 419		
Volatiles in Soil						
Benzene	µg/Kg		75.8	45.9 - 117		
Bromodichloromethane	µg/Kg		148	75.0 - 231		
Bromoform	µg/Kg		88.5	41.4 - 147		
Carbon tetrachloride	µg/Kg		85.4	35.5 - 141		
Chlorobenzene	µg/Kg		103	49.2 - 155		
Chlorodibromomethane	µg/Kg		141	74.9 - 217		
Chloroform	µg/Kg		127	70.8 - 181		
1,2-Dichlorobenzene	µg/Kg		198	53.4 - 313		

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ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)						
1,3-Dichlorobenzene	µg/Kg		141	46.1 - 216		
1,4-Dichlorobenzene	µg/Kg		124	33.4 - 191		
1,1-Dichloroethane	µg/Kg		37.9	21.4 - 55.9		
1,2-Dichloroethane	µg/Kg		71.5	42.6 - 109		
Ethylbenzene	µg/Kg		72.2	36.0 - 116		
Methylene chloride	µg/Kg		91.3	40.3 - 147		
4-Methyl-2-pentanone (MIBK)	µg/Kg		97.4	31.9 - 166		
1,1,1,2-Tetrachloroethane	µg/Kg		55.6	29.0 - 89.0		
1,1,2,2-Tetrachloroethane	µg/Kg		102	45.9 - 158		
Tetrachloroethylene	µg/Kg		88.5	37.4 - 130		
Toluene	µg/Kg		96.4	48.4 - 150		
1,1,1-Trichloroethane	µg/Kg		170	99.0 - 243		
Trichloroethylene	µg/Kg		69.3	34.5 - 110		
Total, Xylenes	µg/Kg		176	65.8 - 290		
Herbicides in Soil						
2,4-D	µg/Kg		390	0.00 - 822		
Dicamba	µg/Kg		212	0.00 - 392		
Disulfoton	µg/Kg		434	0.00 - 683		
2,4,5-T	µg/Kg		296	0.00 - 686		
2,4,5-TP (Silvex)	µg/Kg		139	0.00 - 284		
PCBs in Soil						
Aroclor 1016	mg/Kg		0.00			
Aroclor 1221	mg/Kg		0.00			
Aroclor 1232	mg/Kg		0.00			
Aroclor 1242	mg/Kg		0.00			
Aroclor 1248	mg/Kg		19.0	4.00 - 29.8		
Aroclor 1254	mg/Kg		0.00			
Aroclor 1260	mg/Kg		0.00			

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SOIL-33 Final NELAC Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 03/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
CA Volatiles in Soil						
Benzene	µg/Kg		3790	2770 - 5490		
Bromodichloromethane	µg/Kg		7420	4070 - 11500		
Bromoform	µg/Kg		4420	2320 - 7040		
Carbon tetrachloride	µg/Kg		4270	2450 - 6390		
Chlorobenzene	µg/Kg		5160	3940 - 6780		
Chlorodibromomethane	µg/Kg		7030	3990 - 10600		
Chloroform	µg/Kg		6330	4070 - 8810		
1,2-Dichlorobenzene	µg/Kg		9900	6010 - 13700		
1,3-Dichlorobenzene	µg/Kg		7060	4310 - 10400		
1,4-Dichlorobenzene	µg/Kg		6190	4310 - 8050		
1,1-Dichloroethane	µg/Kg		1890	966 - 2930		
1,2-Dichloroethane	µg/Kg		3580	2210 - 5490		
Ethylbenzene	µg/Kg		3610	2440 - 5410		
Methylene chloride	µg/Kg		4570	1800 - 7280		
4-Methyl-2-pentanone (MIBK)	µg/Kg		4650	2410 - 7110		
1,1,1,2-Tetrachloroethane	µg/Kg		2780	1850 - 4170		
1,1,2,2-Tetrachloroethane	µg/Kg		5110	2770 - 7810		
Tetrachloroethylene	µg/Kg		4420	3040 - 6120		
Toluene	µg/Kg		4820	3360 - 6660		
1,1,1-Trichloroethane	µg/Kg		8520	4500 - 13100		
Trichloroethylene	µg/Kg		3470	2550 - 4860		
Total, Xylenes	µg/Kg		8810	6330 - 12600		

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SOIL-34 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 EPA ID: KS00902 State ID: Report Issued: 06/26/01

InterLaB™ SOIL Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Value for the Organic Proficiency Testing Standards is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation as applicable. The ERA Assigned Value for the Inorganic Proficiency Testing Standards is equal to the maximum amount of the parameter available in the standard by applicable EPA methodologies. All NELAC parameters not added to a standard are given an assigned Value of "0", per the guidance issued by the NELAC Board of Directors, on December 14, 2000. Non-NELAC parameters not added to a standard may be given an Assigned Value of less than a minimum verified concentration as determined in the background soil for applicable EPA methodologies.

The Acceptance Limits are established per the NELAC PT program criteria, state specific PT program requirements or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For a further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside the Acceptance Limits.
No Evaluation	=	Reported Value that can not be evaluated.

The Method Description is the method the laboratory reported to ERA.

InterLaB™ SOIL Study Discussion:

ERA Soil Proficiency Testing Study 34 has been reviewed by ERA Senior Management and certified compliant with the requirements of the National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program, Chapter 2, June 2000.

Per the requirements of the NELAC Proficiency Testing Program, the SOIL-34 results were examined for any study anomalies. A full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the SOIL-34 standards met the acceptance criteria contained in the NELAC Proficiency Testing Program, Chapter 2, June 2000.

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's review of the data, one study anomoliy was found and all Acceptance Limits for the InterLaB™ Soil study, SOIL-34, were calculated based on the NELAC, Proficiency Testing Program, Chapter 2, June 2000, as applicable.

The Soil Proficiency Testing Program study SOIL-34 report shall not be reproduced except in its entirety and without the full written permission of the participating laboratory. The report must not be used by the participating laboratory to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding Soil Study, SOIL-34, please contact Shawn Kassner, InterLaB™ Program Coordinator, or Curtis Wood, Quality Assurance Manager, at 1-800-372-0122.

NVLAP® Lab code: 200366-0

- Study Discussion Concerning Soil-34: Semivolatiles Standard - Endrin aldehyde, Endrin ketone

During the review of the Soil-34 study data, ERA observed a failure rate of 25.0% for endrin aldehyde, and 20.0% for endrin ketone in the Soil Semivolatiles standard. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'.

During our analysis of the data we observed several laboratories reporting values greater than 100 mg/Kg for endrin aldehyde and endrin ketone. These two breakdown products of endrin are produced in the injection port during analysis on the gas chromatograph. Most EPA methodologies allow a 15% degradation rate for endrin to either endrin aldehyde or endrin ketone. Endrin was contained in the Semivolatiles in Soil standard at a concentration of 167 mg/Kg. The maximum degradation concentration for endrin to endrin aldehyde and/or endrin ketone would not produce levels greater than 100 mg/Kg. ERA's Semivolatiles in Soil standard is manufactured to contain base/neutral, acid, and pesticide components to mimic a true environmental soil sample. The base/neutral components in the Semivolatiles in Soil standard include halogenated hydrocarbons, which are detectable by a gas chromatograph connected to an Electron Capture Detector. As specified in EPA methodologies a column clean-up procedure is required to separate the pesticides from other halogenated hydrocarbons. Without a column clean-up laboratories run the risk of reporting false positives during the pesticides analysis. ERA believes the values reported above 100 ug/Kg for endrin aldehyde and endrin ketone in Soil-34 study are the result of misidentification of the BN halogenated hydrocarbons.

If you have any questions please feel free to call ERA's Organic Chemistry Department at 1-800-372-0122.

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Study: **SOIL34**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **ANALYTICAL MANAGEMENT LABS**

Report Type: **Complete**

Report Method: **Method A**

5540 Marshall St., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-34 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 06/26/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil						
Aluminum	mg/Kg	4612	6040	1460 - 11500	Acceptable	6010B
Antimony	mg/Kg	129	190	0.00 - 281	Acceptable	6010B
Arsenic	mg/Kg	234	206	132 - 258	Acceptable	6010B
Barium	mg/Kg	413	392	278 - 503	Acceptable	6010B
Beryllium	mg/Kg	80.1	80.1	52.8 - 97.5	Acceptable	6010B
Boron	mg/Kg		122	10.2 - 122		
Cadmium	mg/Kg	84.5	77.5	49.2 - 88.8	Acceptable	6010B
Calcium	mg/Kg	3532	3310	2200 - 4510	Acceptable	6010B
Chromium	mg/Kg	91.6	85.2	51.8 - 112	Acceptable	6010B
Cobalt	mg/Kg	92.4	103	57.7 - 104	Acceptable	6010B
Copper	mg/Kg	132	127	89.4 - 163	Acceptable	6010B
Iron	mg/Kg	9673	11800	1180 - 21800	Acceptable	6010B
Lead	mg/Kg	116	97.0	65.3 - 125	Acceptable	6010B
Magnesium	mg/Kg	1855	1960	1200 - 2840	Acceptable	6010B
Manganese	mg/Kg	492	465	293 - 603	Acceptable	6010B
Mercury	mg/Kg	8.9	9.11	4.27 - 14.5	Acceptable	7471A
Molybdenum	mg/Kg	86.8	89.7	51.4 - 110	Acceptable	6010B
Nickel	mg/Kg	101	93.6	60.4 - 110	Acceptable	6010B
Potassium	mg/Kg	1721	1820	731 - 3030	Acceptable	6010B
Selenium	mg/Kg	124	124	72.6 - 161	Acceptable	6010B
Silver	mg/Kg	16.8	170	28.3 - 262	Not Acceptable	6010B
Sodium	mg/Kg	327	316	80.0 - 590	Acceptable	6010B
Strontium	mg/Kg		76.9	46.3 - 115		
Thallium	mg/Kg	119	111	56.5 - 150	Acceptable	6010B
Tin	mg/Kg		168	53.2 - 168		
Titanium	mg/Kg		282	0.00 - 696		
Vanadium	mg/Kg	162	162	98.9 - 211	Acceptable	6010B
Zinc	mg/Kg	314	291	193 - 353	Acceptable	6010B
Cyanide in Soil						
Cyanide, total	mg/Kg	141.9	152	0.00 - 248	Acceptable	9010B
Reactive Cyanide	mg/Kg		< 25.0			

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SOIL-34 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 06/26/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Hexavalent Chromium in Soil						
Chromium VI	mg/Kg	78.5	130	14.1 - 156	Acceptable	7196A

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-34 Final Complete Report

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15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 06/26/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil						
Acenaphthene	µg/Kg	5320	4600	1200 - 5820	Acceptable	8270C
Acenaphthylene	µg/Kg	7573	6250	1070 - 7030	Not Acceptable	8270C
Aniline	µg/Kg		6770	0.00 - 6770		
Anthracene	µg/Kg	7015	6830	1640 - 7700	Acceptable	8270C
Benzidine	µg/Kg		0.00			
Benzoic acid	µg/Kg		0.00			
Benzo(a)anthracene	µg/Kg	1253	1120	422 - 1520	Acceptable	8270C
Benzo(b)fluoranthene	µg/Kg	2528	2400	546 - 3530	Acceptable	8270C
Benzo(k)fluoranthene	µg/Kg	3794	3840	989 - 5350	Acceptable	8270C
Benzo(g,h,i)perylene	µg/Kg	3001	3040	527 - 4370	Acceptable	8270C
Benzo(a)pyrene	µg/Kg	6213	6340	1620 - 6940	Acceptable	8270C
Benzyl alcohol	µg/Kg		0.00			
4-Bromophenyl-phenylether	µg/Kg	3262	3350	1100 - 4520	Acceptable	8270C
Butylbenzylphthalate	µg/Kg	9678	7560	1420 - 11000	Acceptable	8270C
Carbazole	µg/Kg	< 100	0.00		Acceptable	8270C
4-Chloroaniline	µg/Kg	< 100	0.00		Acceptable	8270C
bis(2-Chloroethoxy)methane	µg/Kg	< 100	0.00		Acceptable	8270C
bis(2-Chloroethyl)ether	µg/Kg	< 100	0.00		Acceptable	8270C
bis(2-Chloroisopropyl)ether	µg/Kg	< 100	0.00		Acceptable	8270C
4-Chloro-3-methylphenol	µg/Kg	< 100	0.00		Acceptable	8270C
1-Chloronaphthalene	µg/Kg		0.00			
2-Chloronaphthalene	µg/Kg	< 100	0.00		Acceptable	8270C
2-Chlorophenol	µg/Kg	5893	6240	590 - 6950	Acceptable	8270C
4-Chlorophenyl-phenylether	µg/Kg	< 100	0.00		Acceptable	8270C
Chrysene	µg/Kg	5889	5010	1440 - 6800	Acceptable	8270C
Dibenz(a,h)anthracene	µg/Kg	1421	1120	174 - 1650	Acceptable	8270C
Dibenzofuran	µg/Kg	9703	8190	1990 - 10500	Acceptable	8270C
Di-n-butylphthalate	µg/Kg	5999	4540	1020 - 6620	Acceptable	8270C
1,2-Dichlorobenzene	µg/Kg	< 100	0.00		Acceptable	8270C
1,3-Dichlorobenzene	µg/Kg	< 100	0.00		Acceptable	8270C
1,4-Dichlorobenzene	µg/Kg	< 100	0.00		Acceptable	8270C
3,3'-Dichlorobenzidine	µg/Kg		0.00			
2,4-Dichlorophenol	µg/Kg	< 100	0.00		Acceptable	8270C
2,6-Dichlorophenol	µg/Kg	< 100	0.00		Acceptable	8270C
Diethylphthalate	µg/Kg	4516	3810	1160 - 5100	Acceptable	8270C
2,4-Dimethylphenol	µg/Kg	< 100	0.00		Acceptable	8270C
Dimethylphthalate	µg/Kg	6298	5470	1640 - 6900	Acceptable	8270C
4,6-Dinitro-2-methylphenol	µg/Kg	< 100	0.00		Acceptable	8270C
2,4-Dinitrophenol	µg/Kg	< 100	0.00		Acceptable	8270C
2,4-Dinitrotoluene	µg/Kg	< 100	0.00		Acceptable	8270C
2,6-Dinitrotoluene	µg/Kg	9231	7760	2430 - 9690	Acceptable	8270C

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SOIL-34 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 06/26/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)						
Di-n-octylphthalate	µg/Kg	13944	11300	770 - 17800	Acceptable	8270C
bis(2-Ethylhexyl)phthalate	µg/Kg	3863	3590	971 - 5130	Acceptable	8270C
Fluoranthene	µg/Kg	9576	7900	2370 - 10500	Acceptable	8270C
Fluorene	µg/Kg	< 100	0.00		Acceptable	8270C
Hexachlorobenzene	µg/Kg	7845	6490	1620 - 8700	Acceptable	8270C
Hexachlorobutadiene	µg/Kg	< 100	0.00		Acceptable	8270C
Hexachlorocyclopentadiene	µg/Kg	< 100	4090	0.00 - 4090	Acceptable	8270C
Hexachloroethane	µg/Kg	1657	2030	0.00 - 2330	Acceptable	8270C
Indeno(1,2,3-cd)pyrene	µg/Kg	< 100	0.00		Acceptable	8270C
Isophorone	µg/Kg	8428	6070	690 - 8070	Not Acceptable	8270C
2-Methylnaphthalene	µg/Kg	< 100	0.00		Acceptable	8270C
2-Methylphenol	µg/Kg	< 100	0.00		Acceptable	8270C
3-Methylphenol	µg/Kg		0.00			
4-Methylphenol	µg/Kg	1926	4830	0.00 - 4830	Acceptable	8270C
Naphthalene	µg/Kg	3189	3510	602 - 4240	Acceptable	8270C
2-Nitroaniline	µg/Kg	< 100	0.00		Acceptable	8270C
3-Nitroaniline	µg/Kg	< 100	0.00		Acceptable	8270C
4-Nitroaniline	µg/Kg	< 100	0.00		Acceptable	8270C
Nitrobenzene	µg/Kg	8766	8560	1160 - 10500	Acceptable	8270C
2-Nitrophenol	µg/Kg	7985	7950	850 - 9070	Acceptable	8270C
3-Nitrophenol	µg/Kg		0.00			
4-Nitrophenol	µg/Kg	< 100	0.00		Acceptable	8270C
N-Nitrosodimethylamine	µg/Kg	< 100	0.00		Acceptable	8270C
N-Nitrosodiphenylamine	µg/Kg		0.00			
N-Nitroso-di-n-propylamine	µg/Kg	14652	14800	1980 - 19000	Acceptable	8270C
Pentachlorophenol	µg/Kg	< 100	0.00		Acceptable	8270C
Phenanthrene	µg/Kg	6327	5120	1580 - 6940	Acceptable	8270C
Phenol	µg/Kg	4953	4940	521 - 5760	Acceptable	8270C
Pyrene	µg/Kg	< 100	0.00		Acceptable	8270C
Pyridine	µg/Kg		0.00			
1,2,4-Trichlorobenzene	µg/Kg	8157	8090	900 - 9780	Acceptable	8270C
2,4,5-Trichlorophenol	µg/Kg	7391	7170	970 - 8170	Acceptable	8270C
2,4,6-Trichlorophenol	µg/Kg	9221	9310	1220 - 9980	Acceptable	8270C
Additional Base / Neutrals and Acids in Soil						
2-Amino-1-methylbenzene (o-touluidine)	µg/Kg		0.00			
Benzotrichloride	µg/Kg		0.00			
Benzyl chloride	µg/Kg		0.00			
bis(2-methoxyethyl)phthalate	µg/Kg		0.00			
2-Chlorophenyl-4-nitrophenylether	µg/Kg		0.00			
3-Chlorophenyl-4-nitrophenylether	µg/Kg		0.00			
4-Chlorophenyl-4-nitrophenylether	µg/Kg		0.00			

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ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 06/26/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil (Cont.)						
Diamylphthalate	µg/Kg		0.00			
2,4-Dibromophenyl-4-nitrophenylether	µg/Kg		0.00			
2,4-Dichlorophenyl-3-methyl-4-nitrophenylether	µg/Kg		0.00			
2,3-Dichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,4-Dichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,5-Dichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,6-Dichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
3,4-Dichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
3,5-Dichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
Dicyclohexylphthalate	µg/Kg		0.00			
4-Nitrophenylphenylether	µg/Kg		0.00			
Dinonylphthalate	µg/Kg		0.00			
Hexachlorocyclohexane	µg/Kg		0.00			
Hexyl-2-ethylhexylphthalate	µg/Kg		0.00			
Maleic anhydride	µg/Kg		0.00			
Pentachlorobenzene	µg/Kg		0.00			
Pentachlorohexane	µg/Kg		0.00			
Tetrachlorobenzenes	µg/Kg		0.00			
1,2,3,4-Tetrachlorobenzene	µg/Kg		0.00			
1,2,3,5-Tetrachlorobenzene	µg/Kg		0.00			
1,2,4,5-Tetrachlorobenzene	µg/Kg		0.00			
2,3,4,5-Tetrachlorophenol	µg/Kg		0.00			
2,3,4,6-Tetrachlorophenol	µg/Kg		0.00			
2,3,5,6-Tetrachlorophenol	µg/Kg		0.00			
1,2,3-Trichlorobenzene	µg/Kg		0.00			
1,3,5-Trichlorobenzene	µg/Kg		0.00			
2,3,4-Trichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,3,5-Trichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,3,6-Trichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,4,5-Trichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
2,4,6-Trichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
3,4,5-Trichlorophenyl-4-nitrophenylether	µg/Kg		0.00			
Pesticides in Soil						
Aldrin	µg/Kg	346.5	360	106 - 529	Acceptable	8081A
alpha-BHC	µg/Kg	157.3	145	37.9 - 222	Acceptable	8081A
beta-BHC	µg/Kg	384.2	373	38.8 - 545	Acceptable	8081A
delta-BHC	µg/Kg	< 5	0.00		Acceptable	8081A
gamma-BHC(Lindane)	µg/Kg	415.7	410	96.9 - 593	Acceptable	8081A
alpha-Chlordane	µg/Kg	285.2	309	79.2 - 449	Acceptable	8081A
gamma-Chlordane	µg/Kg	< 5	0.00		Acceptable	8081A
4,4'-DDD	µg/Kg	178.7	146	21.0 - 255	Acceptable	8081A

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 06/26/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Pesticides in Soil (Cont.)						
4,4'-DDE	µg/Kg	332.5	351	85.6 - 540	Acceptable	8081A
4,4'-DDT	µg/Kg	203.5	189	19.5 - 323	Acceptable	8081A
Dieldrin	µg/Kg	215.3	183	51.4 - 281	Acceptable	8081A
Endrin	µg/Kg	315.9	267	54.6 - 455	Acceptable	8081A
Endrin aldehyde	µg/Kg	< 50	12.1	0.00 - 27.3	Not Acceptable	8081A
Endrin ketone	µg/Kg	< 50	7.07	0.00 - 18.9	Not Acceptable	8081A
Endosulfan I	µg/Kg	205.8	235	31.0 - 361	Acceptable	8081A
Endosulfan II	µg/Kg	258	407	29.9 - 448	Acceptable	8081A
Endosulfan sulfate	µg/Kg	511.6	446	0.00 - 807	Acceptable	8081A
Heptachlor	µg/Kg	296.2	290	94.2 - 410	Acceptable	8081A
Heptachlor epoxide	µg/Kg	< 50	28.0	0.00 - 59.6	Acceptable	8081A
Methoxychlor	µg/Kg	276.7	206	0.00 - 423	Acceptable	8081A
Chlordane in Soil						
Chlordane, Technical	µg/Kg	293	343	44.8 - 497	Acceptable	EPA 8081A
Toxaphene in Soil						
Toxaphene	µg/Kg	411	430	0.00 - 714	Acceptable	EPA 8081A
Volatiles in Soil						
Acetone	µg/Kg	81.1	61.3	0.00 - 275	Acceptable	8260B
Acetonitrile	µg/Kg		0.00			
Acrolein	µg/Kg		0.00			
Benzene	µg/Kg	55.6	60.7	29.7 - 88.5	Acceptable	8260B
Bromodichloromethane	µg/Kg	54.7	63.6	27.7 - 97.9	Acceptable	8260B
Bromoform	µg/Kg	117.4	116	32.2 - 192	Acceptable	8260B
Bromomethane	µg/Kg	< 10	0.00		Acceptable	8260B
2-Butanone (MEK)	µg/Kg	< 10	19.7	0.00 - 70.0	Acceptable	8260B
Carbon disulfide	µg/Kg	< 10	0.00		Acceptable	8260B
Carbon tetrachloride	µg/Kg	33	38.8	14.3 - 60.7	Acceptable	8260B
Chlorobenzene	µg/Kg	136.5	140	66.4 - 206	Acceptable	8260B
Chlorodibromomethane	µg/Kg	34.3	38.5	12.2 - 61.6	Acceptable	8260B
Chloroethane	µg/Kg	< 10	0.00		Acceptable	8260B
2-Chloroethylvinylether	µg/Kg		0.00			
Chloroform	µg/Kg	62.1	71.2	37.0 - 104	Acceptable	8260B
Chloromethane	µg/Kg	< 10	0.00		Acceptable	8260B
1,2-Dibromo-3-chloropropane	µg/Kg		0.00			
1,2-Dibromoethane (EDB)	µg/Kg	< 10	0.00		Acceptable	8260B
Dibromomethane	µg/Kg	< 10	0.00		Acceptable	8260B
1,2-Dichlorobenzene	µg/Kg	64.7	72.2	23.7 - 111	Acceptable	8260B
1,3-Dichlorobenzene	µg/Kg	81.0	89.2	27.9 - 137	Acceptable	8260B
1,4-Dichlorobenzene	µg/Kg	86.6	92.9	29.5 - 141	Acceptable	8260B
Dichlorodifluoromethane	µg/Kg	< 10	0.00		Acceptable	8260B
1,1-Dichloroethane	µg/Kg	59.4	70.0	33.1 - 108	Acceptable	8260B

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-34 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 06/26/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)						
1,2-Dichloroethane	µg/Kg	45.1	49.2	21.0 - 75.8	Acceptable	8260B
1,1-Dichloroethylene	µg/Kg	< 10	0.00		Acceptable	8260B
cis-1,2-Dichloroethylene	µg/Kg	< 10	0.00		Acceptable	8260B
trans-1,2-Dichloroethylene	µg/Kg	< 10	0.00		Acceptable	8260B
1,2-Dichloropropane	µg/Kg	< 10	0.00		Acceptable	8260B
cis-1,3-Dichloropropylene	µg/Kg	< 10	0.00		Acceptable	8260B
trans-1,3-Dichloropropylene	µg/Kg	< 10	0.00		Acceptable	8260B
Ethylbenzene	µg/Kg	46.2	50.1	21.2 - 79.7	Acceptable	8260B
2-Hexanone	µg/Kg	< 10	0.00		Acceptable	8260B
Methylene chloride	µg/Kg	122.8	135	41.5 - 201	Acceptable	8260B
4-Methyl-2-pentanone (MIBK)	µg/Kg	100.9	97.4	8.60 - 171	Acceptable	8260B
Styrene	µg/Kg	< 10	0.00		Acceptable	8260B
1,1,1,2-Tetrachloroethane	µg/Kg	88.2	93.4	43.6 - 141	Acceptable	8260B
1,1,2,2-Tetrachloroethane	µg/Kg	143.7	148	51.8 - 234	Acceptable	8260B
Tetrachloroethylene	µg/Kg	128.1	133	41.8 - 206	Acceptable	8260B
Toluene	µg/Kg	58.5	57.9	28.8 - 87.8	Acceptable	8260B
1,1,1-Trichloroethane	µg/Kg	45.8	53.5	24.4 - 80.2	Acceptable	8260B
1,1,2-Trichloroethane	µg/Kg	76.0	77.4	34.3 - 118	Acceptable	8260B
Trichloroethylene	µg/Kg	132.7	147	67.6 - 210	Acceptable	8260B
Trichlorofluoromethane	µg/Kg		0.00			
1,2,3-Trichloropropane	µg/Kg		84.7	8.90 - 152		
Vinyl acetate	µg/Kg		0.00			
Vinyl chloride	µg/Kg	< 10	0.00		Acceptable	8260B
Total, Xylenes	µg/Kg	154.0	140	55.2 - 227	Acceptable	8260B
PCBs in Soil						
Aroclor 1016	mg/Kg	< 1	0.00		Acceptable	EPA 8082
Aroclor 1221	mg/Kg	< 1	0.00		Acceptable	EPA 8082
Aroclor 1232	mg/Kg	< 1	0.00		Acceptable	EPA 8082
Aroclor 1242	mg/Kg	12.02	14.1	1.75 - 21.3	Acceptable	EPA 8082
Aroclor 1248	mg/Kg	< 1	0.00		Acceptable	EPA 8082
Aroclor 1254	mg/Kg	< 1	0.00		Acceptable	EPA 8082
Aroclor 1260	mg/Kg	< 1	0.00		Acceptable	EPA 8082

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Study: **SOIL34**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **ANALYTICAL MANAGEMENT LABS**

Report Type: **NELAC**

Report Method: **Method A**

5540 Marshall St., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-34 Final NELAC Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 06/26/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil						
Aluminum	mg/Kg	4612	6040	1460 - 11500	Acceptable	6010B
Antimony	mg/Kg	129	190	0.00 - 281	Acceptable	6010B
Arsenic	mg/Kg	234	206	132 - 258	Acceptable	6010B
Barium	mg/Kg	413	392	278 - 503	Acceptable	6010B
Beryllium	mg/Kg	80.1	80.1	52.8 - 97.5	Acceptable	6010B
Boron	mg/Kg		122	10.2 - 122		
Cadmium	mg/Kg	84.5	77.5	49.2 - 88.8	Acceptable	6010B
Calcium	mg/Kg	3532	3310	2200 - 4510	Acceptable	6010B
Chromium	mg/Kg	91.6	85.2	51.8 - 112	Acceptable	6010B
Cobalt	mg/Kg	92.4	103	57.7 - 104	Acceptable	6010B
Copper	mg/Kg	132	127	89.4 - 163	Acceptable	6010B
Iron	mg/Kg	9673	11800	1180 - 21800	Acceptable	6010B
Lead	mg/Kg	116	97.0	65.3 - 125	Acceptable	6010B
Magnesium	mg/Kg	1855	1960	1200 - 2840	Acceptable	6010B
Manganese	mg/Kg	492	465	293 - 603	Acceptable	6010B
Mercury	mg/Kg	8.9	9.11	4.27 - 14.5	Acceptable	7471A
Molybdenum	mg/Kg	86.8	89.7	51.4 - 110	Acceptable	6010B
Nickel	mg/Kg	101	93.6	60.4 - 110	Acceptable	6010B
Potassium	mg/Kg	1721	1820	731 - 3030	Acceptable	6010B
Selenium	mg/Kg	124	124	72.6 - 161	Acceptable	6010B
Silver	mg/Kg	16.8	170	28.3 - 262	Not Acceptable	6010B
Sodium	mg/Kg	327	316	80.0 - 590	Acceptable	6010B
Thallium	mg/Kg	119	111	56.5 - 150	Acceptable	6010B
Tin	mg/Kg		168	53.2 - 168		
Vanadium	mg/Kg	162	162	98.9 - 211	Acceptable	6010B
Zinc	mg/Kg	314	291	193 - 353	Acceptable	6010B
Cyanide in Soil						
Cyanide, total	mg/Kg	141.9	152	0.00 - 248	Acceptable	9010B

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-34 Final NELAC Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 06/26/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Hexavalent Chromium in Soil						
Chromium VI	mg/Kg	78.5	130	14.1 - 156	Acceptable	7196A

NVLAP® Lab code: 200386-0

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 06/26/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil						
Acenaphthene	µg/Kg	5320	4600	1200 - 5820	Acceptable	8270C
Acenaphthylene	µg/Kg	7573	6250	1070 - 7030	Not Acceptable	8270C
Anthracene	µg/Kg	7015	6830	1640 - 7700	Acceptable	8270C
Benzo(a)anthracene	µg/Kg	1253	1120	422 - 1520	Acceptable	8270C
Benzo(b)fluoranthene	µg/Kg	2528	2400	546 - 3530	Acceptable	8270C
Benzo(k)fluoranthene	µg/Kg	3794	3840	989 - 5350	Acceptable	8270C
Benzo(g,h,i)perylene	µg/Kg	3001	3040	527 - 4370	Acceptable	8270C
Benzo(a)pyrene	µg/Kg	6213	6340	1620 - 6940	Acceptable	8270C
4-Bromophenyl-phenylether	µg/Kg	3262	3350	1100 - 4520	Acceptable	8270C
Butylbenzylphthalate	µg/Kg	9678	7560	1420 - 11000	Acceptable	8270C
bis(2-Chloroethoxy)methane	µg/Kg	< 100	0.00		Acceptable	8270C
bis(2-Chloroisopropyl)ether	µg/Kg	< 100	0.00		Acceptable	8270C
4-Chloro-3-methylphenol	µg/Kg	< 100	0.00		Acceptable	8270C
2-Chloronaphthalene	µg/Kg	< 100	0.00		Acceptable	8270C
2-Chlorophenol	µg/Kg	5893	6240	590 - 6950	Acceptable	8270C
4-Chlorophenyl-phenylether	µg/Kg	< 100	0.00		Acceptable	8270C
Chrysene	µg/Kg	5889	5010	1440 - 6800	Acceptable	8270C
Dibenz(a,h)anthracene	µg/Kg	1421	1120	174 - 1650	Acceptable	8270C
Dibenzofuran	µg/Kg	9703	8190	1990 - 10500	Acceptable	8270C
Di-n-butylphthalate	µg/Kg	5999	4540	1020 - 6620	Acceptable	8270C
2,4-Dichlorophenol	µg/Kg	< 100	0.00		Acceptable	8270C
Diethylphthalate	µg/Kg	4516	3810	1160 - 5100	Acceptable	8270C
2,4-Dimethylphenol	µg/Kg	< 100	0.00		Acceptable	8270C
Dimethylphthalate	µg/Kg	6298	5470	1640 - 6900	Acceptable	8270C
4,6-Dinitro-2-methylphenol	µg/Kg	< 100	0.00		Acceptable	8270C
2,4-Dinitrophenol	µg/Kg	< 100	0.00		Acceptable	8270C
2,4-Dinitrotoluene	µg/Kg	< 100	0.00		Acceptable	8270C
2,6-Dinitrotoluene	µg/Kg	9231	7760	2430 - 9690	Acceptable	8270C
Di-n-octylphthalate	µg/Kg	13944	11300	770 - 17800	Acceptable	8270C
bis(2-Ethylhexyl)phthalate	µg/Kg	3863	3590	971 - 5130	Acceptable	8270C
Fluoranthene	µg/Kg	9576	7900	2370 - 10500	Acceptable	8270C
Fluorene	µg/Kg	< 100	0.00		Acceptable	8270C
Hexachlorobenzene	µg/Kg	7845	6490	1620 - 8700	Acceptable	8270C
Hexachlorobutadiene	µg/Kg	< 100	0.00		Acceptable	8270C
Hexachlorocyclopentadiene	µg/Kg	< 100	4090	0.00 - 4090	Acceptable	8270C
Hexachloroethane	µg/Kg	1657	2030	0.00 - 2330	Acceptable	8270C
Indeno(1,2,3-cd)pyrene	µg/Kg	< 100	0.00		Acceptable	8270C
Isophorone	µg/Kg	8428	6070	690 - 8070	Not Acceptable	8270C
2-Methylphenol	µg/Kg	< 100	0.00		Acceptable	8270C
Naphthalene	µg/Kg	3189	3510	602 - 4240	Acceptable	8270C
Nitrobenzene	µg/Kg	8766	8560	1160 - 10500	Acceptable	8270C

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SOIL-34 Final NELAC Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 06/26/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)						
2-Nitrophenol	µg/Kg	7985	7950	850 - 9070	Acceptable	8270C
4-Nitrophenol	µg/Kg	< 100	0.00		Acceptable	8270C
N-Nitroso-di-n-propylamine	µg/Kg	14652	14800	1980 - 19000	Acceptable	8270C
Pentachlorophenol	µg/Kg	< 100	0.00		Acceptable	8270C
Phenanthrene	µg/Kg	6327	5120	1580 - 6940	Acceptable	8270C
Phenol	µg/Kg	4953	4940	521 - 5760	Acceptable	8270C
Pyrene	µg/Kg	< 100	0.00		Acceptable	8270C
1,2,4-Trichlorobenzene	µg/Kg	8157	8090	900 - 9780	Acceptable	8270C
2,4,5-Trichlorophenol	µg/Kg	7391	7170	970 - 8170	Acceptable	8270C
2,4,6-Trichlorophenol	µg/Kg	9221	9310	1220 - 9980	Acceptable	8270C
Pesticides in Soil						
Aldrin	µg/Kg	346.5	360	106 - 529	Acceptable	8081A
alpha-BHC	µg/Kg	157.3	145	37.9 - 222	Acceptable	8081A
beta-BHC	µg/Kg	384.2	373	38.8 - 545	Acceptable	8081A
delta-BHC	µg/Kg	< 5	0.00		Acceptable	8081A
gamma-BHC(Lindane)	µg/Kg	415.7	410	96.9 - 593	Acceptable	8081A
4,4'-DDD	µg/Kg	178.7	146	21.0 - 255	Acceptable	8081A
4,4'-DDE	µg/Kg	332.5	351	85.6 - 540	Acceptable	8081A
4,4'-DDT	µg/Kg	203.5	189	19.5 - 323	Acceptable	8081A
Dieldrin	µg/Kg	215.3	183	51.4 - 281	Acceptable	8081A
Endrin	µg/Kg	315.9	267	54.6 - 455	Acceptable	8081A
Endrin aldehyde	µg/Kg	< 50	12.1	0.00 - 27.3	Not Acceptable	8081A
Endosulfan I	µg/Kg	205.8	235	31.0 - 361	Acceptable	8081A
Endosulfan II	µg/Kg	258	407	29.9 - 448	Acceptable	8081A
Endosulfan sulfate	µg/Kg	511.6	446	0.00 - 807	Acceptable	8081A
Heptachlor	µg/Kg	296.2	290	94.2 - 410	Acceptable	8081A
Heptachlor epoxide	µg/Kg	< 50	28.0	0.00 - 59.6	Acceptable	8081A
Methoxychlor	µg/Kg	276.7	206	0.00 - 423	Acceptable	8081A
Chlordane in Soil						
Chlordane, Technical	µg/Kg	293	343	44.8 - 497	Acceptable	EPA 8081A
Toxaphene in Soil						
Toxaphene	µg/Kg	411	430	0.00 - 714	Acceptable	EPA 8081A
Volatiles in Soil						
Benzene	µg/Kg	55.6	60.7	29.7 - 88.5	Acceptable	8260B
Bromodichloromethane	µg/Kg	54.7	63.6	27.7 - 97.9	Acceptable	8260B
Bromoform	µg/Kg	117.4	116	32.2 - 192	Acceptable	8260B
Carbon tetrachloride	µg/Kg	33	38.8	14.3 - 60.7	Acceptable	8260B
Chlorobenzene	µg/Kg	136.5	140	66.4 - 206	Acceptable	8260B
Chlorodibromomethane	µg/Kg	34.3	38.5	12.2 - 61.6	Acceptable	8260B
Chloroform	µg/Kg	62.1	71.2	37.0 - 104	Acceptable	8260B
1,2-Dichlorobenzene	µg/Kg	64.7	72.2	23.7 - 111	Acceptable	8260B

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SOIL-34 Final NELAC Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 06/26/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)						
1,3-Dichlorobenzene	µg/Kg	81.0	89.2	27.9 - 137	Acceptable	8260B
1,4-Dichlorobenzene	µg/Kg	86.6	92.9	29.5 - 141	Acceptable	8260B
1,1-Dichloroethane	µg/Kg	59.4	70.0	33.1 - 108	Acceptable	8260B
1,2-Dichloroethane	µg/Kg	45.1	49.2	21.0 - 75.8	Acceptable	8260B
Ethylbenzene	µg/Kg	46.2	50.1	21.2 - 79.7	Acceptable	8260B
Methylene chloride	µg/Kg	122.8	135	41.5 - 201	Acceptable	8260B
4-Methyl-2-pentanone (MIBK)	µg/Kg	100.9	97.4	8.60 - 171	Acceptable	8260B
1,1,1,2-Tetrachloroethane	µg/Kg	88.2	93.4	43.6 - 141	Acceptable	8260B
1,1,2,2-Tetrachloroethane	µg/Kg	143.7	148	51.8 - 234	Acceptable	8260B
Tetrachloroethylene	µg/Kg	128.1	133	41.8 - 206	Acceptable	8260B
Toluene	µg/Kg	58.5	57.9	28.8 - 87.8	Acceptable	8260B
1,1,1-Trichloroethane	µg/Kg	45.8	53.5	24.4 - 80.2	Acceptable	8260B
Trichloroethylene	µg/Kg	132.7	147	67.6 - 210	Acceptable	8260B
Total, Xylenes	µg/Kg	154.0	140	55.2 - 227	Acceptable	8260B
PCBs in Soil						
Aroclor 1016	mg/Kg	< 1	0.00		Acceptable	EPA 8082
Aroclor 1221	mg/Kg	< 1	0.00		Acceptable	EPA 8082
Aroclor 1232	mg/Kg	< 1	0.00		Acceptable	EPA 8082
Aroclor 1242	mg/Kg	12.02	14.1	1.75 - 21.3	Acceptable	EPA 8082
Aroclor 1248	mg/Kg	< 1	0.00		Acceptable	EPA 8082
Aroclor 1254	mg/Kg	< 1	0.00		Acceptable	EPA 8082
Aroclor 1260	mg/Kg	< 1	0.00		Acceptable	EPA 8082

NVLAP®
Lab code: 200388-0

SOIL-34 Definitions & Study Discussion

ERA Laboratory Code: A5230-01A EPA ID: KS00902 State ID: E-10254 Report Issued: 06/26/01

InterLaB™ SOIL Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Value for the Organic Proficiency Testing Standards is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation as applicable. The ERA Assigned Value for the Inorganic Proficiency Testing Standards is equal to the maximum amount of the parameter available in the standard by applicable EPA methodologies. All NELAC parameters not added to a standard are given an assigned Value of "0", per the guidance issued by the NELAC Board of Directors, on December 14, 2000. Non-NELAC parameters not added to a standard may be given an Assigned Value of less than a minimum verified concentration as determined in the background soil for applicable EPA methodologies.

The Acceptance Limits are established per the NELAC PT program criteria, state specific PT program requirements or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For a further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable = Reported Value falls within the Acceptance Limits.

Not Acceptable = Reported Value falls outside the Acceptance Limits.

No Evaluation = Reported Value that can not be evaluated.

The Method Description is the method the laboratory reported to ERA.

InterLaB™ SOIL Study Discussion:

ERA Soil Proficiency Testing Study 34 has been reviewed by ERA Senior Management and certified compliant with the requirements of the National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program, Chapter 2, June 2000.

Per the requirements of the NELAC Proficiency Testing Program, the SOIL-34 results were examined for any study anomalies. A full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the SOIL-34 standards met the acceptance criteria contained in the NELAC Proficiency Testing Program, Chapter 2, June 2000.

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's review of the data, one study anomoliy was found and all Acceptance Limits for the InterLaB™ Soil study, SOIL-34, were calculated based on the NELAC, Proficiency Testing Program, Chapter 2, June 2000, as applicable.

The Soil Proficiency Testing Program study SOIL-34 report shall not be reproduced except in its entirety and without the full written permission of the participating laboratory. The report must not be used by the participating laboratory to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding Soil Study, SOIL-34, please contact Shawn Kassner, InterLaB™ Program Coordinator, or Curtis Wood, Quality Assurance Manager, at 1-800-372-0122.

NVLAP® Lab code: 200388-0

- Study Discussion Concerning Soil-34: Semivolatiles Standard - Endrin aldehyde, Endrin ketone

During the review of the Soil-34 study data, ERA observed a failure rate of 25.0% for endrin aldehyde, and 20.0% for endrin ketone in the Soil Semivolatiles standard. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'.

During our analysis of the data we observed several laboratories reporting values greater than 100 mg/Kg for endrin aldehyde and endrin ketone. These two breakdown products of endrin are produced in the injection port during analysis on the gas chromatograph. Most EPA methodologies allow a 15% degradation rate for endrin to either endrin aldehyde or endrin ketone. Endrin was contained in the Semivolatiles in Soil standard at a concentration of 167 mg/Kg. The maximum degradation concentration for endrin to endrin aldehyde and/or endrin ketone would not produce levels greater than 100 mg/Kg. ERA's Semivolatiles in Soil standard is manufactured to contain base/neutral, acid, and pesticide components to mimic a true environmental soil sample. The base/neutral components in the Semivolatiles in Soil standard include halogenated hydrocarbons, which are detectable by a gas chromatograph connected to an Electron Capture Detector. As specified in EPA methodologies a column clean-up procedure is required to separate the pesticides from other halogenated hydrocarbons. Without a column clean-up laboratories run the risk of reporting false positives during the pesticides analysis. ERA believes the values reported above 100 ug/Kg for endrin aldehyde and endrin ketone in Soil-34 study are the result of misidentification of the BN halogenated hydrocarbons.

If you have any questions please feel free to call ERA's Organic Chemistry Department at 1-800-372-0122.

NVLAP® Lab code: 200386-0

ENVIRONMENTAL
RESOURCE ASSOCIATES®

Study: **SOIL34**

ERA Laboratory Code: **A5230-01A**

Laboratory Name: **ANALYTICAL MANAGEMENT LABS**

Report Type: **Complete**

Report Method: **Method A**

5540 Marshall St., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-34 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 06/26/01**
State ID: E-10254
ERA Laboratory Code: A5230-01A

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Herbicides in Soil						
2,4-D	µg/Kg	334	390	0.00 - 645	Acceptable	8151
Dalapon	µg/Kg	< 42	0.00		Acceptable	8151
2,4-DB	µg/Kg	1140	1180	0.00 - 1840	Acceptable	8151
Dicamba	µg/Kg	348	425	0.00 - 715	Acceptable	8151
Dinoseb	µg/Kg	166	1010	0.00 - 1460	Acceptable	8151
Disulfoton	µg/Kg		471	0.00 - 1750		
MCPP	µg/Kg	< 1670	0.00		Acceptable	8151
Pentachlorophenol	µg/Kg		988	0.00 - 1810		
2,4,5-T	µg/Kg	233	310	0.00 - 505	Acceptable	8151
2,4,5-TP (Silvex)	µg/Kg	350	418	0.00 - 695	Acceptable	8151

The Reported Values shown were received after the limits were published.

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ENVIRONMENTAL
RESOURCE ASSOCIATES®

Study: **SOIL34**

ERA Laboratory Code: **A5230-01A**

Laboratory Name: **ANALYTICAL MANAGEMENT LABS**

Report Type: **NELAC**

Report Method: **Method A**

5540 Marshall St., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-34 Final NELAC Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 06/26/01**
State ID: E-10254
ERA Laboratory Code: A5230-01A

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Herbicides in Soil						
2,4-D	µg/Kg	334	390	0.00 - 645	Acceptable	8151
Dicamba	µg/Kg	348	425	0.00 - 715	Acceptable	8151
Disulfoton	µg/Kg		471	0.00 - 1750		
2,4,5-T	µg/Kg	233	310	0.00 - 505	Acceptable	8151
2,4,5-TP (Silvex)	µg/Kg	350	418	0.00 - 695	Acceptable	8151

The Reported Values shown were received after the limits were published.

NVLAP® Lab code: 200388-0

SOIL-36 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 EPA ID: KS00902 State ID: Report Issued: 12/28/01

InterLaB™ SOIL Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Value for the Organic Proficiency Testing Standards is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation as applicable. The ERA Assigned Value for the Inorganic Proficiency Testing Standards is equal to the maximum amount of the parameter available in the standard by applicable EPA methodologies. All NELAC parameters not added to a standard are given an assigned Value of "0", per the guidance issued by the NELAC Board of Directors, on December 14, 2000. Non-NELAC parameters not added to a standard may be given an Assigned Value of less than a minimum verified concentration as determined in the background soil for applicable EPA methodologies.

The Acceptance Limits are established per the NELAC PT program criteria, state specific PT program requirements or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For a further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable = Reported Value falls within the Acceptance Limits.

Not Acceptable = Reported Value falls outside the Acceptance Limits.

No Evaluation = Reported Value cannot be evaluated.

The Method Description is the method the laboratory reported to ERA.

InterLaB™ SOIL Study Discussion:

ERA Soil Proficiency Testing Study 36 has been reviewed by ERA Senior Management and certified compliant with the requirements of the National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program, Chapter 2, June 2000.

Per the requirements of the NELAC Proficiency Testing Program, the Soil-36 results were examined for any study anomalies. A full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the Soil-36 standards met the acceptance criteria contained in the NELAC Proficiency Testing Program, Chapter 2, June 2000.

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's review of the data, one study anomoly was found. The anomaly is listed on the next page. All Acceptance Limits for the Soil-36 Study were calculated based on the NELAC, Proficiency Testing Program, Chapter 2, June 2000, as applicable.

The Soil Proficiency Testing Program Study, Soil-36, report shall not be reproduced except in its entirety and without the full written permission of the participating laboratory. The report must not be used by the participating laboratory to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding Soil Study, Soil-36, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

- Study Discussion - Volatiles in Soil: 2-Hexanone

2-Hexanone was found in the background for the soil matrix for the Volatiles in Soil sample for the Soil-36 Study. The concentration of 2-Hexanone was analytically determined to be 2.90 mg/kg. This compound is not on the field of testing for any national or state program. The acceptance limits were calculated based on the NELAC, Proficiency Testing Program, Chapter 2, June 2000. If you have any further questions please feel free to call ERA's Organic Chemistry Department at 1-800-2372-0122.

NVLAP® Lab code: 200386-0

ENVIRONMENTAL
RESOURCE ASSOCIATES®

Study: **SOIL36**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **ANALYTICAL MANAGEMENT LABS**

Report Type: **Complete**

Report Method: **Method A**

5540 Marshall St., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-36 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 12/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil						
Aluminum	mg/kg	5400	5810	1340 - 11400	Acceptable	EPA 6010B
Antimony	mg/kg	260	270	0.00 - 313	Acceptable	EPA 6010B
Arsenic	mg/kg	318	299	173 - 389	Acceptable	EPA 6010B
Barium	mg/kg	560	537	334 - 746	Acceptable	EPA 6010B
Beryllium	mg/kg	48.2	49.4	28.4 - 65.2	Acceptable	EPA 6010B
Boron	mg/kg		267	80.9 - 267		
Cadmium	mg/kg	55.3	56.6	31.8 - 70.6	Acceptable	EPA 6010B
Calcium	mg/kg	3490	3040	2070 - 4650	Acceptable	EPA 6010B
Chromium	mg/kg	69.7	66.0	39.9 - 88.2	Acceptable	EPA 6010B
Cobalt	mg/kg	132	143	80.3 - 164	Acceptable	EPA 6010B
Copper	mg/kg	175	167	100 - 234	Acceptable	EPA 6010B
Iron	mg/kg	12200	10800	1140 - 21100	Acceptable	EPA 6010B
Lead	mg/kg	93.4	85.7	54.3 - 117	Acceptable	EPA 6010B
Magnesium	mg/kg	1980	1830	1170 - 2870	Acceptable	EPA 6010B
Manganese	mg/kg	476	441	295 - 613	Acceptable	EPA 6010B
Mercury	mg/kg	20.0	16.9	6.35 - 27.7	Acceptable	EPA 7471A
Molybdenum	mg/kg	135	128	69.3 - 165	Acceptable	EPA 6010B
Nickel	mg/kg	65.8	62.3	38.3 - 78.3	Acceptable	EPA 6010B
Potassium	mg/kg	1750	1680	900 - 2820	Acceptable	EPA 6010B
Selenium	mg/kg	149	136	69.8 - 180	Acceptable	EPA 6010B
Silver	mg/kg	103	98.1	41.8 - 140	Acceptable	EPA 6010B
Sodium	mg/kg	282	258	0.00 - 630	Acceptable	EPA 6010B
Strontium	mg/kg		102	66.2 - 154		
Thallium	mg/kg	92.5	98.6	44.2 - 138	Acceptable	EPA 6010B
Tin	mg/kg		197	31.6 - 222		
Titanium	mg/kg		338	0.00 - 691		
Vanadium	mg/kg	133	130	76.4 - 174	Acceptable	EPA 6010B
Zinc	mg/kg	166	147	91.3 - 205	Acceptable	EPA 6010B

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-36 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 12/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Hexavalent Chromium in Soil						
Chromium VI	mg/kg	117	267	5.10 - 277	Acceptable	EPA 7196A

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-36 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 12/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil						
Acenaphthene	µg/kg	5690	7090	1520 - 8720	Acceptable	EPA 8270C
Acenaphthylene	µg/kg	3830	5360	499 - 6060	Acceptable	EPA 8270C
Aniline	µg/kg		0.00			
Anthracene	µg/kg	1900	3270	710 - 3530	Acceptable	EPA 8270C
Benzidine	µg/kg		0.00			
Benzoic acid	µg/kg		0.00			
Benzo(a)anthracene	µg/kg	2210	2660	746 - 3670	Acceptable	EPA 8270C
Benzo(b)fluoranthene	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Benzo(k)fluoranthene	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Benzo(g,h,i)perylene	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Benzo(a)pyrene	µg/kg	2240	4140	707 - 4490	Acceptable	EPA 8270C
Benzyl alcohol	µg/kg		0.00			
4-Bromophenyl-phenylether	µg/kg	2340	3360	895 - 4650	Acceptable	EPA 8270C
Butylbenzylphthalate	µg/kg	6580	7630	2050 - 10300	Acceptable	EPA 8270C
Carbazole	µg/kg	< 10	0.00		Acceptable	EPA 8270C
4-Chloroaniline	µg/kg	< 10	0.00		Acceptable	EPA 8270C
bis(2-Chloroethoxy)methane	µg/kg	6050	8130	1060 - 10200	Acceptable	EPA 8270C
bis(2-Chloroethyl)ether	µg/kg	< 10	0.00		Acceptable	EPA 8270C
bis(2-Chloroisopropyl)ether	µg/kg	< 10	0.00		Acceptable	EPA 8270C
4-Chloro-3-methylphenol	µg/kg	2720	4170	918 - 4420	Acceptable	EPA 8270C
1-Chloronaphthalene	µg/kg	< 10	0.00		Acceptable	EPA 8270C
2-Chloronaphthalene	µg/kg	6170	8210	1130 - 10400	Acceptable	EPA 8270C
2-Chlorophenol	µg/kg	5250	9600	400 - 9940	Acceptable	EPA 8270C
4-Chlorophenyl-phenylether	µg/kg	5430	5970	1530 - 7950	Acceptable	EPA 8270C
Chrysene	µg/kg	857	980	270 - 1500	Acceptable	EPA 8270C
Dibenz(a,h)anthracene	µg/kg	2400	3220	826 - 4470	Acceptable	EPA 8270C
Dibenzofuran	µg/kg	1940	2330	640 - 2980	Acceptable	EPA 8270C
Di-n-butylphthalate	µg/kg	5660	6200	1270 - 8710	Acceptable	EPA 8270C
1,2-Dichlorobenzene	µg/kg	3670	5960	0.00 - 6800	Acceptable	EPA 8270C
1,3-Dichlorobenzene	µg/kg	< 10	0.00		Acceptable	EPA 8270C
1,4-Dichlorobenzene	µg/kg	< 10	0.00		Acceptable	EPA 8270C
3,3'-Dichlorobenzidine	µg/kg	< 10	0.00		Acceptable	EPA 8270C
2,4-Dichlorophenol	µg/kg	1890	3170	368 - 3330	Acceptable	EPA 8270C
2,6-Dichlorophenol	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Diethylphthalate	µg/kg	4650	5170	1090 - 6830	Acceptable	EPA 8270C
2,4-Dimethylphenol	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Dimethylphthalate	µg/kg	6440	8240	2050 - 10100	Acceptable	EPA 8270C
4,6-Dinitro-2-methylphenol	µg/kg	< 10	0.00		Acceptable	EPA 8270C
2,4-Dinitrophenol	µg/kg	< 10	0.00		Acceptable	EPA 8270C
2,4-Dinitrotoluene	µg/kg	2050	3010	666 - 3830	Acceptable	EPA 8270C
2,6-Dinitrotoluene	µg/kg	< 10	0.00		Acceptable	EPA 8270C

NVLAP® Lab code: 200386-0

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 12/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)						
Di-n-octylphthalate	µg/kg	7090	7000	1320 - 10700	Acceptable	EPA 8270C
bis(2-Ethylhexyl)phthalate	µg/kg	6540	7380	1360 - 10700	Acceptable	EPA 8270C
Fluoranthene	µg/kg	7120	7310	1800 - 9780	Acceptable	EPA 8270C
Fluorene	µg/kg	2210	2650	698 - 3420	Acceptable	EPA 8270C
Hexachlorobenzene	µg/kg	2140	2730	777 - 3660	Acceptable	EPA 8270C
Hexachlorobutadiene	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Hexachlorocyclopentadiene	µg/kg	< 10	8810	0.00 - 8810	Acceptable	EPA 8270C
Hexachloroethane	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Indeno(1,2,3-cd)pyrene	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Isophorone	µg/kg	2520	3020	267 - 4110	Acceptable	EPA 8270C
2-Methylnaphthalene	µg/kg	< 10	0.00		Acceptable	EPA 8270C
2-Methylphenol	µg/kg	2050	7150	0.00 - 7150	Acceptable	EPA 8270C
3-Methylphenol	µg/kg	2970	6060	0.00 - 7570	Acceptable	EPA 8270C
4-Methylphenol	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Naphthalene	µg/kg	1520	2180	138 - 2860	Acceptable	EPA 8270C
2-Nitroaniline	µg/kg	< 10	0.00		Acceptable	EPA 8270C
3-Nitroaniline	µg/kg	< 10	0.00		Acceptable	EPA 8270C
4-Nitroaniline	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Nitrobenzene	µg/kg	5570	8620	1110 - 9990	Acceptable	EPA 8270C
2-Nitrophenol	µg/kg	4330	7100	590 - 8210	Acceptable	EPA 8270C
3-Nitrophenol	µg/kg	< 10	0.00		Acceptable	EPA 8270C
4-Nitrophenol	µg/kg	3430	7930	0.00 - 10300	Acceptable	EPA 8270C
N-Nitrosodimethylamine	µg/kg		0.00			
N-Nitrosodiphenylamine	µg/kg	< 10	0.00		Acceptable	EPA 8270C
N-Nitroso-di-n-propylamine	µg/kg	4680	7690	360 - 10100	Acceptable	EPA 8270C
Pentachlorophenol	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Phenanthrene	µg/kg	1150	1430	454 - 2030	Acceptable	EPA 8270C
Phenol	µg/kg	7430	12100	840 - 12800	Acceptable	EPA 8270C
Pyrene	µg/kg	2400	3280	768 - 4690	Acceptable	EPA 8270C
Pyridine	µg/kg	< 10	0.00		Acceptable	EPA 8270C
1,2,4-Trichlorobenzene	µg/kg	3450	5080	351 - 6130	Acceptable	EPA 8270C
2,4,5-Trichlorophenol	µg/kg	2370	4280	462 - 4760	Acceptable	EPA 8270C
2,4,6-Trichlorophenol	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Additional Base / Neutrals and Acids in Soil						
2-Amino-1-methylbenzene (o-touluidine)	µg/kg		0.00			
Benzotrichloride	µg/kg		0.00			
Benzyl chloride	µg/kg		0.00			
bis(2-methoxyethyl)phthalate	µg/kg		0.00			
2-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
3-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
4-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			

NVLAP® Lab code: 200386-0

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 12/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil (Cont.)						
Diamylphthalate	µg/kg		0.00			
2,4-Dibromophenyl-4-nitrophenylether	µg/kg		0.00			
2,4-Dichlorophenyl-3-methyl-4-nitrophenyl	µg/kg		0.00			
2,3-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
2,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
2,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
2,6-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
3,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
3,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
Dicyclohexylphthalate	µg/kg		0.00			
4-Nitrophenylphenylether	µg/kg		0.00			
Dinonylphthalate	µg/kg		0.00			
Hexachlorocyclohexane	µg/kg		0.00			
Hexyl-2-ethylhexylphthalate	µg/kg		0.00			
Maleic anhydride	µg/kg		0.00			
Pentachlorobenzene	µg/kg		0.00			
Pentachlorohexane	µg/kg		0.00			
Tetrachlorobenzenes	µg/kg		0.00			
1,2,3,4-Tetrachlorobenzene	µg/kg		0.00			
1,2,3,5-Tetrachlorobenzene	µg/kg		0.00			
1,2,4,5-Tetrachlorobenzene	µg/kg		0.00			
2,3,4,5-Tetrachlorophenol	µg/kg		0.00			
2,3,4,6-Tetrachlorophenol	µg/kg		0.00			
2,3,5,6-Tetrachlorophenol	µg/kg		0.00			
1,2,3-Trichlorobenzene	µg/kg		0.00			
1,3,5-Trichlorobenzene	µg/kg		0.00			
2,3,4-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
2,3,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
2,3,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
2,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
2,4,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
3,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
Pesticides in Soil						
Aldrin	µg/kg	179	183	41.1 - 283	Acceptable	EPA 8081A
alpha-BHC	µg/kg	260	149	22.9 - 243	Not Acceptable	EPA 8081A
beta-BHC	µg/kg	355	430	29.0 - 665	Acceptable	EPA 8081A
delta-BHC	µg/kg	< 5	0.00		Acceptable	EPA 8081A
gamma-BHC(Lindane)	µg/kg	401	355	85.3 - 547	Acceptable	EPA 8081A
alpha-Chlordane	µg/kg	196	194	54.2 - 298	Acceptable	EPA 8081A
gamma-Chlordane	µg/kg	128	135	10.8 - 229	Acceptable	EPA 8081A
4,4'-DDD	µg/kg	179	79.0	6.40 - 152	Not Acceptable	EPA 8081A

NVLAP® Lab code: 200386-0

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 12/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Pesticides in Soil (Cont.)						
4,4'-DDE	µg/kg	183	178	16.2 - 302	Acceptable	EPA 8081A
4,4'-DDT	µg/kg	365	308	30.5 - 568	Acceptable	EPA 8081A
Dieldrin	µg/kg	201	197	30.2 - 328	Acceptable	EPA 8081A
Endrin	µg/kg	358	289	30.6 - 527	Acceptable	EPA 8081A
Endrin aldehyde	µg/kg	< 5	102	0.00 - 177	Acceptable	EPA 8081A
Endrin ketone	µg/kg	< 5	91.7	3.10 - 162	Not Acceptable	EPA 8081A
Endosulfan I	µg/kg	< 5	0.00		Acceptable	EPA 8081A
Endosulfan II	µg/kg	< 5	0.00		Acceptable	EPA 8081A
Endosulfan sulfate	µg/kg	153	156	0.00 - 292	Acceptable	EPA 8081A
Heptachlor	µg/kg	316	282	41.3 - 461	Acceptable	EPA 8081A
Heptachlor epoxide	µg/kg	75	70.9	14.8 - 128	Acceptable	EPA 8081A
Methoxychlor	µg/kg	35	221	0.00 - 484	Acceptable	EPA 8081A
Chlordane in Soil						
Chlordane, Technical	µg/kg	233	368	58.9 - 555	Acceptable	EPA 8081A
Toxaphene in Soil						
Toxaphene	µg/kg	351	396	0.00 - 600	Acceptable	EPA 8081A
Volatiles in Soil						
Acetone	µg/kg	126	206	0.00 - 495	Acceptable	EPA 8260B
Acetonitrile	µg/kg		0.00			
Acrolein	µg/kg		0.00			
Benzene	µg/kg	117	116	55.8 - 172	Acceptable	EPA 8260B
Bromodichloromethane	µg/kg	74.8	71.4	31.4 - 112	Acceptable	EPA 8260B
Bromoform	µg/kg	83.7	74.8	28.2 - 118	Acceptable	EPA 8260B
Bromomethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
2-Butanone (MEK)	µg/kg	55.3	183	0.00 - 338	Acceptable	EPA 8260B
Carbon disulfide	µg/kg	< 10	0.00		Acceptable	EPA 8260B
Carbon tetrachloride	µg/kg	85.7	88.7	37.5 - 135	Acceptable	EPA 8260B
Chlorobenzene	µg/kg	123	115	63.3 - 159	Acceptable	EPA 8260B
Chlorodibromomethane	µg/kg	71.7	66.9	30.7 - 99.1	Acceptable	EPA 8260B
Chloroethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
2-Chloroethylvinylether	µg/kg	< 10	0.00		Acceptable	EPA 8260B
Chloroform	µg/kg	45.8	42.4	22.2 - 63.6	Acceptable	EPA 8260B
Chloromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
1,2-Dibromo-3-chloropropane	µg/kg		42.2	6.40 - 74.2		
1,2-Dibromoethane (EDB)	µg/kg	58.3	54.2	24.7 - 80.1	Acceptable	EPA 8260B
Dibromomethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
1,2-Dichlorobenzene	µg/kg	41.6	38.3	13.9 - 60.3	Acceptable	EPA 8260B
1,3-Dichlorobenzene	µg/kg	110	107	30.8 - 169	Acceptable	EPA 8260B
1,4-Dichlorobenzene	µg/kg	174	166	56.9 - 253	Acceptable	EPA 8260B
Dichlorodifluoromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
1,1-Dichloroethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B

NVLAP® Lab code: 200386-0

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 12/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)						
1,2-Dichloroethane	µg/kg	75.7	71.9	28.5 - 111	Acceptable	EPA 8260B
1,1-Dichloroethylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
cis-1,2-Dichloroethylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
trans-1,2-Dichloroethylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
1,2-Dichloropropane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
cis-1,3-Dichloropropylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
trans-1,3-Dichloropropylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
Ethylbenzene	µg/kg	51.0	48.7	23.5 - 74.5	Acceptable	EPA 8260B
2-Hexanone	µg/kg	< 10	2.90	0.00 - 9.53	Acceptable	EPA 8260B
Methylene chloride	µg/kg	87.1	87.9	13.3 - 150	Acceptable	EPA 8260B
4-Methyl-2-pentanone (MIBK)	µg/kg	157	150	37.4 - 237	Acceptable	EPA 8260B
Styrene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
1,1,1,2-Tetrachloroethane	µg/kg	100	90.2	47.2 - 132	Acceptable	EPA 8260B
1,1,2,2-Tetrachloroethane	µg/kg	195	169	53.9 - 280	Acceptable	EPA 8260B
Tetrachloroethylene	µg/kg	47.6	48.8	15.5 - 78.5	Acceptable	EPA 8260B
Toluene	µg/kg	51.1	50.5	27.1 - 74.3	Acceptable	EPA 8260B
1,1,1-Trichloroethane	µg/kg	134	136	52.8 - 211	Acceptable	EPA 8260B
1,1,2-Trichloroethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
Trichloroethylene	µg/kg	127	124	47.6 - 190	Acceptable	EPA 8260B
Trichlorofluoromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
1,2,3-Trichloropropane	µg/kg	< 10	87.5	25.3 - 147	Not Acceptable	EPA 8260B
Vinyl acetate	µg/kg		0.00			
Vinyl chloride	µg/kg	< 25	0.00		Acceptable	EPA 8260B
Total, Xylenes	µg/kg	258	239	114 - 370	Acceptable	EPA 8260B
PCBs in Soil						
Aroclor 1016	mg/kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1221	mg/kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1232	mg/kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1242	mg/kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1248	mg/kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1254	mg/kg	11.2	14.8	4.39 - 21.0	Acceptable	EPA 8082
Aroclor 1260	mg/kg	< 0.5	0.00		Acceptable	EPA 8082

NVLAP® Lab code: 200386-0

ENVIRONMENTAL
RESOURCE ASSOCIATES®

Study: **SOIL36**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **ANALYTICAL MANAGEMENT LABS**

Report Type: **NELAC**

Report Method: **Method A**

5540 Marshall St., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-36 Final NELAC Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 12/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil						
Aluminum	mg/kg	5400	5810	1340 - 11400	Acceptable	EPA 6010B
Antimony	mg/kg	260	270	0.00 - 313	Acceptable	EPA 6010B
Arsenic	mg/kg	318	299	173 - 389	Acceptable	EPA 6010B
Barium	mg/kg	560	537	334 - 746	Acceptable	EPA 6010B
Beryllium	mg/kg	48.2	49.4	28.4 - 65.2	Acceptable	EPA 6010B
Boron	mg/kg		267	80.9 - 267		
Cadmium	mg/kg	55.3	56.6	31.8 - 70.6	Acceptable	EPA 6010B
Calcium	mg/kg	3490	3040	2070 - 4650	Acceptable	EPA 6010B
Chromium	mg/kg	69.7	66.0	39.9 - 88.2	Acceptable	EPA 6010B
Cobalt	mg/kg	132	143	80.3 - 164	Acceptable	EPA 6010B
Copper	mg/kg	175	167	100 - 234	Acceptable	EPA 6010B
Iron	mg/kg	12200	10800	1140 - 21100	Acceptable	EPA 6010B
Lead	mg/kg	93.4	85.7	54.3 - 117	Acceptable	EPA 6010B
Magnesium	mg/kg	1980	1830	1170 - 2870	Acceptable	EPA 6010B
Manganese	mg/kg	476	441	295 - 613	Acceptable	EPA 6010B
Mercury	mg/kg	20.0	16.9	6.35 - 27.7	Acceptable	EPA 7471A
Molybdenum	mg/kg	135	128	69.3 - 165	Acceptable	EPA 6010B
Nickel	mg/kg	65.8	62.3	38.3 - 78.3	Acceptable	EPA 6010B
Potassium	mg/kg	1750	1680	900 - 2820	Acceptable	EPA 6010B
Selenium	mg/kg	149	136	69.8 - 180	Acceptable	EPA 6010B
Silver	mg/kg	103	98.1	41.8 - 140	Acceptable	EPA 6010B
Sodium	mg/kg	282	258	0.00 - 630	Acceptable	EPA 6010B
Thallium	mg/kg	92.5	98.6	44.2 - 138	Acceptable	EPA 6010B
Tin	mg/kg		197	31.6 - 222		
Vanadium	mg/kg	133	130	76.4 - 174	Acceptable	EPA 6010B
Zinc	mg/kg	166	147	91.3 - 205	Acceptable	EPA 6010B

NVLAP® Lab code: 200386-0

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 Report Issued: 12/28/01
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Hexavalent Chromium in Soil						
Chromium VI	mg/kg	117	267	5.10 - 277	Acceptable	EPA 7196A

NVLAP® Labcode: 200386-0

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 12/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil						
Acenaphthene	µg/kg	5690	7090	1520 - 8720	Acceptable	EPA 8270C
Acenaphthylene	µg/kg	3830	5360	499 - 6060	Acceptable	EPA 8270C
Anthracene	µg/kg	1900	3270	710 - 3530	Acceptable	EPA 8270C
Benzo(a)anthracene	µg/kg	2210	2660	746 - 3670	Acceptable	EPA 8270C
Benzo(b)fluoranthene	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Benzo(k)fluoranthene	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Benzo(g,h,i)perylene	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Benzo(a)pyrene	µg/kg	2240	4140	707 - 4490	Acceptable	EPA 8270C
4-Bromophenyl-phenylether	µg/kg	2340	3360	895 - 4650	Acceptable	EPA 8270C
Butylbenzylphthalate	µg/kg	6580	7630	2050 - 10300	Acceptable	EPA 8270C
bis(2-Chloroethoxy)methane	µg/kg	6050	8130	1060 - 10200	Acceptable	EPA 8270C
bis(2-Chloroisopropyl)ether	µg/kg	< 10	0.00		Acceptable	EPA 8270C
4-Chloro-3-methylphenol	µg/kg	2720	4170	918 - 4420	Acceptable	EPA 8270C
2-Chloronaphthalene	µg/kg	6170	8210	1130 - 10400	Acceptable	EPA 8270C
2-Chlorophenol	µg/kg	5250	9600	400 - 9940	Acceptable	EPA 8270C
4-Chlorophenyl-phenylether	µg/kg	5430	5970	1530 - 7950	Acceptable	EPA 8270C
Chrysene	µg/kg	857	980	270 - 1500	Acceptable	EPA 8270C
Dibenz(a,h)anthracene	µg/kg	2400	3220	826 - 4470	Acceptable	EPA 8270C
Dibenzofuran	µg/kg	1940	2330	640 - 2980	Acceptable	EPA 8270C
Di-n-butylphthalate	µg/kg	5660	6200	1270 - 8710	Acceptable	EPA 8270C
2,4-Dichlorophenol	µg/kg	1890	3170	368 - 3330	Acceptable	EPA 8270C
Diethylphthalate	µg/kg	4650	5170	1090 - 6830	Acceptable	EPA 8270C
2,4-Dimethylphenol	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Dimethylphthalate	µg/kg	6440	8240	2050 - 10100	Acceptable	EPA 8270C
4,6-Dinitro-2-methylphenol	µg/kg	< 10	0.00		Acceptable	EPA 8270C
2,4-Dinitrophenol	µg/kg	< 10	0.00		Acceptable	EPA 8270C
2,4-Dinitrotoluene	µg/kg	2050	3010	666 - 3830	Acceptable	EPA 8270C
2,6-Dinitrotoluene	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Di-n-octylphthalate	µg/kg	7090	7000	1320 - 10700	Acceptable	EPA 8270C
bis(2-Ethylhexyl)phthalate	µg/kg	6540	7380	1360 - 10700	Acceptable	EPA 8270C
Fluoranthene	µg/kg	7120	7310	1800 - 9780	Acceptable	EPA 8270C
Fluorene	µg/kg	2210	2650	698 - 3420	Acceptable	EPA 8270C
Hexachlorobenzene	µg/kg	2140	2730	777 - 3660	Acceptable	EPA 8270C
Hexachlorobutadiene	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Hexachlorocyclopentadiene	µg/kg	< 10	8810	0.00 - 8810	Acceptable	EPA 8270C
Hexachloroethane	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Indeno(1,2,3-cd)pyrene	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Isophorone	µg/kg	2520	3020	267 - 4110	Acceptable	EPA 8270C
2-Methylphenol	µg/kg	2050	7150	0.00 - 7150	Acceptable	EPA 8270C
Naphthalene	µg/kg	1520	2180	138 - 2860	Acceptable	EPA 8270C
Nitrobenzene	µg/kg	5570	8620	1110 - 9990	Acceptable	EPA 8270C

NVLAP® Lab code: 200386-0

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 12/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)						
2-Nitrophenol	µg/kg	4330	7100	590 - 8210	Acceptable	EPA 8270C
4-Nitrophenol	µg/kg	3430	7930	0.00 - 10300	Acceptable	EPA 8270C
N-Nitroso-di-n-propylamine	µg/kg	4680	7690	360 - 10100	Acceptable	EPA 8270C
Pentachlorophenol	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Phenanthrene	µg/kg	1150	1430	454 - 2030	Acceptable	EPA 8270C
Phenol	µg/kg	7430	12100	840 - 12800	Acceptable	EPA 8270C
Pyrene	µg/kg	2400	3280	768 - 4690	Acceptable	EPA 8270C
1,2,4-Trichlorobenzene	µg/kg	3450	5080	351 - 6130	Acceptable	EPA 8270C
2,4,5-Trichlorophenol	µg/kg	2370	4280	462 - 4760	Acceptable	EPA 8270C
2,4,6-Trichlorophenol	µg/kg	< 10	0.00		Acceptable	EPA 8270C
Pesticides in Soil						
Aldrin	µg/kg	179	183	41.1 - 283	Acceptable	EPA 8081A
alpha-BHC	µg/kg	260	149	22.9 - 243	Not Acceptable	EPA 8081A
beta-BHC	µg/kg	355	430	29.0 - 665	Acceptable	EPA 8081A
delta-BHC	µg/kg	< 5	0.00		Acceptable	EPA 8081A
gamma-BHC(Lindane)	µg/kg	401	355	85.3 - 547	Acceptable	EPA 8081A
4,4'-DDD	µg/kg	179	79.0	6.40 - 152	Not Acceptable	EPA 8081A
4,4'-DDE	µg/kg	183	178	16.2 - 302	Acceptable	EPA 8081A
4,4'-DDT	µg/kg	365	308	30.5 - 568	Acceptable	EPA 8081A
Dieldrin	µg/kg	201	197	30.2 - 328	Acceptable	EPA 8081A
Endrin	µg/kg	358	289	30.6 - 527	Acceptable	EPA 8081A
Endrin aldehyde	µg/kg	< 5	102	0.00 - 177	Acceptable	EPA 8081A
Endosulfan I	µg/kg	< 5	0.00		Acceptable	EPA 8081A
Endosulfan II	µg/kg	< 5	0.00		Acceptable	EPA 8081A
Endosulfan sulfate	µg/kg	153	156	0.00 - 292	Acceptable	EPA 8081A
Heptachlor	µg/kg	316	282	41.3 - 461	Acceptable	EPA 8081A
Heptachlor epoxide	µg/kg	75	70.9	14.8 - 128	Acceptable	EPA 8081A
Methoxychlor	µg/kg	35	221	0.00 - 484	Acceptable	EPA 8081A
Chlordane in Soil						
Chlordane, Technical	µg/kg	233	368	58.9 - 555	Acceptable	EPA 8081A
Toxaphene in Soil						
Toxaphene	µg/kg	351	396	0.00 - 600	Acceptable	EPA 8081A
Volatiles in Soil						
Benzene	µg/kg	117	116	55.8 - 172	Acceptable	EPA 8260B
Bromodichloromethane	µg/kg	74.8	71.4	31.4 - 112	Acceptable	EPA 8260B
Bromoform	µg/kg	83.7	74.8	28.2 - 118	Acceptable	EPA 8260B
Carbon tetrachloride	µg/kg	85.7	88.7	37.5 - 135	Acceptable	EPA 8260B
Chlorobenzene	µg/kg	123	115	63.3 - 159	Acceptable	EPA 8260B
Chlorodibromomethane	µg/kg	71.7	66.9	30.7 - 99.1	Acceptable	EPA 8260B
Chloroform	µg/kg	45.8	42.4	22.2 - 63.6	Acceptable	EPA 8260B
1,2-Dichlorobenzene	µg/kg	41.6	38.3	13.9 - 60.3	Acceptable	EPA 8260B

NVLAP®
Lab code: 200386-0

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 12/28/01**
State ID:
ERA Laboratory Code: A5230-01

ERA Standard/Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)						
1,3-Dichlorobenzene	µg/kg	110	107	30.8 - 169	Acceptable	EPA 8260B
1,4-Dichlorobenzene	µg/kg	174	166	56.9 - 253	Acceptable	EPA 8260B
1,1-Dichloroethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
1,2-Dichloroethane	µg/kg	75.7	71.9	28.5 - 111	Acceptable	EPA 8260B
Ethylbenzene	µg/kg	51.0	48.7	23.5 - 74.5	Acceptable	EPA 8260B
Methylene chloride	µg/kg	87.1	87.9	13.3 - 150	Acceptable	EPA 8260B
4-Methyl-2-pentanone (MIBK)	µg/kg	157	150	37.4 - 237	Acceptable	EPA 8260B
1,1,1,2-Tetrachloroethane	µg/kg	100	90.2	47.2 - 132	Acceptable	EPA 8260B
1,1,2,2-Tetrachloroethane	µg/kg	195	169	53.9 - 280	Acceptable	EPA 8260B
Tetrachloroethylene	µg/kg	47.6	48.8	15.5 - 78.5	Acceptable	EPA 8260B
Toluene	µg/kg	51.1	50.5	27.1 - 74.3	Acceptable	EPA 8260B
1,1,1-Trichloroethane	µg/kg	134	136	52.8 - 211	Acceptable	EPA 8260B
Trichloroethylene	µg/kg	127	124	47.6 - 190	Acceptable	EPA 8260B
Total, Xylenes	µg/kg	258	239	114 - 370	Acceptable	EPA 8260B
PCBs in Soil						
Aroclor 1016	mg/kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1221	mg/kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1232	mg/kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1242	mg/kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1248	mg/kg	< 0.5	0.00		Acceptable	EPA 8082
Aroclor 1254	mg/kg	11.2	14.8	4.39 - 21.0	Acceptable	EPA 8082
Aroclor 1260	mg/kg	< 0.5	0.00		Acceptable	EPA 8082

NVLAP® Lab code: 200386-0

ENVIRONMENTAL
RESOURCE ASSOCIATES®
The Industry Standard™

October 15, 2002

Tenkasi Viswanathan
Analytical Management Laboratories
Suite B
15130 South Keeler
Olathe, KS 66062

Dear Tenkasi,

Enclosed is the revised ERA final statistical summary for the InterLaB™ Soil, Soil-39, Proficiency Testing (PT) Program. The revisions requested have been made to the report so that your "less than" results now include the detection limit. As requested a copy of the revised report has been sent to your requested regulatory agency, along with an explanation of what happened.

I apologize for any inconvenience this may have caused. If you have any questions, please do not hesitate to call Shawn Kassner, Proficiency Testing Manager or me at 1-800-372-0122.

Sincerely,

Dana L. Evans
InterLaB™ Proficiency Testing

dle
attachments

SOIL-39 Definitions & Study Discussion

ERA Laboratory Code: A5230-01A EPA ID: KS00902 **Report Issued: 10/15/02**

SOIL Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Value for the Organic Proficiency Testing Standards is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation as applicable. The ERA Assigned Value for the Inorganic Proficiency Testing Standards is equal to the maximum amount of the parameter available in the standard by applicable EPA methodologies. All NELAC parameters not added to a standard are given an assigned Value of "0", per the guidance issued by the NELAC Board of Directors, on December 14, 2000. Non-NELAC parameters not added to a standard may be given an Assigned Value of less than a minimum verified concentration as determined in the background soil for applicable EPA methodologies.

The Acceptance Limits are established per the NELAC PT program criteria, state specific PT program requirements or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For a further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside of the Acceptance Limits.
No Evaluation	=	Reported Value cannot be evaluated.

The Method Description is the method the laboratory reported to ERA.

SOIL Study Discussion:

ERA's Proficiency Testing (PT) Study, Soil-39, has been reviewed by ERA Senior Management and certified compliant with the requirements of the National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program, Chapter 2, June 2000.

The opening date of ERA's Proficiency Testing (PT) Study, Soil-39, was July 29, 2002, and the closing date was September 12, 2002.

Per the requirements of the NELAC Proficiency Testing Program, the Soil-39 results were examined for any study anomalies. A full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the Soil-39 standards met the acceptance criteria contained in the NELAC Proficiency Testing Program, Chapter 2, June 2000.

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's review of the data, no anomalies were noted. All Acceptance Limits for the Soil-39 Study were calculated based on the NELAC Proficiency Testing Program, Chapter 2, June 2000, as applicable.

The Proficiency Testing Study, Soil-39, report shall not be reproduced except in its entirety and without the full written permission of the participating laboratory. The report must not be used by the participating laboratory to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding ERA's Proficiency Testing Study, Soil-39, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

ENVIRONMENTAL
RESOURCE ASSOCIATES®

Study: **SOIL39**

ERA Laboratory Code: **A5230-01A**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method A**

5540 Marshall St., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-39 Final Complete Report

Tenkasi Viswanathan
Analytical Management Labs
Suite B
15130 South Keeler
Olathe, KS 66062
913-829-0101

EPA ID: KS00902
ERA Laboratory Code: A5230-01A
Report Issued: 10/15/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil							
5500	Acenaphthene	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
5505	Acenaphthylene	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
5545	Aniline	µg/kg		0.00			
5555	Anthracene	µg/kg		3320	733 - 3320		
5595	Benzidine	µg/kg		0.00			
5610	Benzoic acid	µg/kg		0.00			
5575	Benzo(a)anthracene	µg/kg		895	309 - 1140		
5585	Benzo(b)fluoranthene	µg/kg		7100	1870 - 9670		
5600	Benzo(k)fluoranthene	µg/kg		3380	790 - 4510		
5590	Benzo(g,h,i)perylene	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
5580	Benzo(a)pyrene	µg/kg		3640	782 - 3640		
5630	Benzyl alcohol	µg/kg		0.00			
5660	4-Bromophenyl-phenylether	µg/kg		7260	2240 - 9620		
5670	Butylbenzylphthalate	µg/kg		2830	829 - 4050		
5680	Carbazole	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
5745	4-Chloroaniline	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
5760	bis(2-Chloroethoxy)methane	µg/kg		3440	843 - 4140		
5765	bis(2-Chloroethyl)ether	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
5780	bis(2-Chloroisopropyl)ether	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
5700	4-Chloro-3-methylphenol	µg/kg		6990	1230 - 7350		
5790	1-Chloronaphthalene	µg/kg		0.00			
5795	2-Chloronaphthalene	µg/kg		1420	261 - 1840		
5800	2-Chlorophenol	µg/kg		8400	750 - 8400		
5825	4-Chlorophenyl-phenylether	µg/kg		7110	2140 - 9040		
5855	Chrysene	µg/kg		1220	403 - 1720		
5895	Dibenz(a,h)anthracene	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
5905	Dibenzofuran	µg/kg		5930	1780 - 7140		
5925	Di-n-butylphthalate	µg/kg		7800	1700 - 10800		
4610	1,2-Dichlorobenzene	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
4615	1,3-Dichlorobenzene	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
4620	1,4-Dichlorobenzene	µg/kg		5900	0.00 - 6780		
5945	3,3'-Dichlorobenzidine	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
6000	2,4-Dichlorophenol	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
6005	2,6-Dichlorophenol	µg/kg		0.00			
6070	Diethylphthalate	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
6130	2,4-Dimethylphenol	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
6135	Dimethylphthalate	µg/kg		8130	1940 - 10300		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

*** Indicated result(s) have been revised.**

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-39 Final Complete Report

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913-829-0101

EPA ID: KS00902
ERA Laboratory Code: A5230-01A
Report Issued: 10/15/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)							
6140	4,6-Dinitro-2-methylphenol	µg/kg		7780	0.00 - 8170		
6175	2,4-Dinitrophenol	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
6185	2,4-Dinitrotoluene	µg/kg		6970	1440 - 8640		
6190	2,6-Dinitrotoluene	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
6200	Di-n-octylphthalate	µg/kg		11000	1120 - 17800		
6255	bis(2-Ethylhexyl)phthalate	µg/kg		4880	920 - 7280		
6265	Fluoranthene	µg/kg		1410	540 - 1800		
6270	Fluorene	µg/kg		1430	461 - 1860		
6275	Hexachlorobenzene	µg/kg		2330	735 - 3050		
4835	Hexachlorobutadiene	µg/kg		9260	0.00 - 12000		
6285	Hexachlorocyclopentadiene	µg/kg	< 330	6840	0.00 - 6840	Acceptable *	EPA 8270C
4840	Hexachloroethane	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
6315	Indeno(1,2,3-cd)pyrene	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
6320	Isophorone	µg/kg		4950	957 - 6240		
6385	2-Methylnaphthalene	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
6400	2-Methylphenol	µg/kg		7610	0.00 - 7610		
6412	3 & 4-Methylphenol	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
5005	Naphthalene	µg/kg		1090	137 - 1390		
6460	2-Nitroaniline	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
6465	3-Nitroaniline	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
6470	4-Nitroaniline	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
5015	Nitrobenzene	µg/kg		7160	610 - 8950		
6490	2-Nitrophenol	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
6495	3-Nitrophenol	µg/kg		0.00			
6500	4-Nitrophenol	µg/kg		7420	0.00 - 8770		
6530	N-Nitrosodimethylamine	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
6535	N-Nitrosodiphenylamine	µg/kg		0.00			
6545	N-Nitroso-di-n-propylamine	µg/kg		9790	1790 - 12400		
6605	Pentachlorophenol	µg/kg		8490	1200 - 8490		
6615	Phenanthrene	µg/kg		967	401 - 1340		
6625	Phenol	µg/kg		13900	1150 - 14800		
6665	Pyrene	µg/kg		1190	374 - 1650		
5095	Pyridine	µg/kg		0.00			
5155	1,2,4-Trichlorobenzene	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
6835	2,4,5-Trichlorophenol	µg/kg	< 330	0.00		Acceptable *	EPA 8270C
6840	2,4,6-Trichlorophenol	µg/kg		8370	1350 - 8370		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

*** Indicated result(s) have been revised.**

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-39 Final Complete Report

Tenkasi Viswanathan
Analytical Management Labs
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913-829-0101

EPA ID: KS00902
ERA Laboratory Code: A5230-01A
Report Issued: 10/15/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil							
5145	2-Amino-1-methylbenzene (o-touluidine)	µg/kg		0.00			
5625	Benzotrichloride	µg/kg		0.00			
5635	Benzyl chloride	µg/kg		0.00			
6350	bis(2-methoxyethyl)phthalate	µg/kg		0.00			
5810	2-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5815	3-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5820	4-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5885	Diamylphthalate	µg/kg		0.00			
5920	2,4-Dibromophenyl-4-nitrophenylether	µg/kg		0.00			
6010	2,4-Dichlorophenyl-3-methyl-4-nitrophenylether	µg/kg		0.00			
6015	2,3-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6020	2,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6025	2,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6030	2,6-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6035	3,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6040	3,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6055	Dicyclohexylphthalate	µg/kg		0.00			
6505	4-Nitrophenylphenylether	µg/kg		0.00			
6195	Dinonylphthalate	µg/kg		0.00			
6280	Hexachlorocyclohexane	µg/kg		0.00			
6305	Hexyl-2-ethylhexylphthalate	µg/kg		0.00			
6335	Maleic anhydride	µg/kg		0.00			
6590	Pentachlorobenzene	µg/kg		0.00			
6595	Pentachlorohexane	µg/kg		0.00			
6717	Tetrachlorobenzenes	µg/kg		0.00			
6705	1,2,3,4-Tetrachlorobenzene	µg/kg		0.00			
6710	1,2,3,5-Tetrachlorobenzene	µg/kg		0.00			
6715	1,2,4,5-Tetrachlorobenzene	µg/kg		0.00			
6730	2,3,4,5-Tetrachlorophenol	µg/kg		0.00			
6735	2,3,4,6-Tetrachlorophenol	µg/kg		0.00			
6740	2,3,5,6-Tetrachlorophenol	µg/kg		0.00			
5150	1,2,3-Trichlorobenzene	µg/kg		0.00			
6800	1,3,5-Trichlorobenzene	µg/kg		0.00			
6845	2,3,4-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6850	2,3,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6855	2,3,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6860	2,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

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Report Issued: 10/15/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil (Cont.)							
6865	2,4,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6870	3,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
Pesticides in Soil							
7025	Aldrin	µg/kg		244	93.1 - 341		
7110	alpha-BHC	µg/kg		168	59.9 - 268		
7115	beta-BHC	µg/kg		147	10.9 - 213		
7105	delta-BHC	µg/kg		189	41.7 - 282		
7120	gamma-BHC(Lindane)	µg/kg		137	22.4 - 216		
7240	alpha-Chlordane	µg/kg	< 10	0.00		Acceptable *	EPA 8081
7245	gamma-Chlordane	µg/kg	< 10	0.00		Acceptable *	EPA 8081
7355	4,4'-DDD	µg/kg		212	77.2 - 303		
7360	4,4'-DDE	µg/kg		294	97.3 - 427		
7365	4,4'-DDT	µg/kg		154	66.2 - 214		
7470	Dieldrin	µg/kg		136	60.0 - 186		
7540	Endrin	µg/kg		146	74.9 - 211		
7530	Endrin aldehyde	µg/kg		270	39.1 - 323		
7535	Endrin ketone	µg/kg		204	61.2 - 305		
7510	Endosulfan I	µg/kg		411	107 - 541		
7515	Endosulfan II	µg/kg		301	70.8 - 379		
7520	Endosulfan sulfate	µg/kg		428	152 - 604		
7685	Heptachlor	µg/kg		123	51.0 - 171		
7690	Heptachlor epoxide	µg/kg		220	92.5 - 354		
7810	Methoxychlor	µg/kg		121	30.8 - 195		
Volatiles in Soil							
4315	Acetone	µg/kg		208	0.00 - 460		
4320	Acetonitrile	µg/kg		0.00			
4325	Acrolein	µg/kg		0.00			
4375	Benzene	µg/kg		90.5	55.1 - 130		
4395	Bromodichloromethane	µg/kg		93.9	54.2 - 141		
4400	Bromoform	µg/kg		94.2	45.5 - 142		
4950	Bromomethane	µg/kg	< 10	0.00		Acceptable *	EPA 8260B
4410	2-Butanone (MEK)	µg/kg		133	20.5 - 216		
4450	Carbon disulfide	µg/kg	< 10	0.00		Acceptable *	EPA 8260B
4455	Carbon tetrachloride	µg/kg		86.4	33.5 - 140		
4475	Chlorobenzene	µg/kg		120	71.1 - 163		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

*** Indicated result(s) have been revised.**

NVLAP® Lab code: 200386-0

Tenkasi Viswanathan
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Suite B
15130 South Keeler
Olathe, KS 66062
913-829-0101

EPA ID: KS00902
ERA Laboratory Code: A5230-01A
Report Issued: 10/15/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)							
4575	Chlorodibromomethane	µg/kg		45.5	24.6 - 67.8		
4485	Chloroethane	µg/kg	< 10	0.00		Acceptable *	EPA 8260B
4500	2-Chloroethylvinylether	µg/kg		0.00			
4505	Chloroform	µg/kg		140	84.2 - 202		
4960	Chloromethane	µg/kg	< 10	0.00		Acceptable *	EPA 8260B
4570	1,2-Dibromo-3-chloropropane	µg/kg		0.00			
4585	1,2-Dibromoethane (EDB)	µg/kg		83.0	54.0 - 115		
4595	Dibromomethane	µg/kg	< 10	0.00		Acceptable *	EPA 8260B
4610	1,2-Dichlorobenzene	µg/kg		30.0	11.4 - 44.1		
4615	1,3-Dichlorobenzene	µg/kg		30.5	10.1 - 44.7		
4620	1,4-Dichlorobenzene	µg/kg		108	37.0 - 155		
4625	Dichlorodifluoromethane	µg/kg	< 10	0.00		Acceptable *	EPA 8260B
4630	1,1-Dichloroethane	µg/kg		66.0	35.1 - 102		
4635	1,2-Dichloroethane	µg/kg		48.3	30.9 - 69.3		
4640	1,1-Dichloroethylene	µg/kg	< 10	0.00		Acceptable *	EPA 8260B
4645	cis-1,2-Dichloroethylene	µg/kg	< 10	0.00		Acceptable *	EPA 8260B
4700	trans-1,2-Dichloroethylene	µg/kg	< 10	0.00		Acceptable *	EPA 8260B
4655	1,2-Dichloropropane	µg/kg	< 10	0.00		Acceptable *	EPA 8260B
4680	cis-1,3-Dichloropropylene	µg/kg	< 10	0.00		Acceptable *	EPA 8260B
4685	trans-1,3-Dichloropropylene	µg/kg	< 10	0.00		Acceptable *	EPA 8260B
4765	Ethylbenzene	µg/kg		110	63.2 - 157		
4860	2-Hexanone	µg/kg	< 10	0.00		Acceptable *	EPA 8260B
4975	Methylene chloride	µg/kg		101	46.2 - 152		
4995	4-Methyl-2-pentanone (MIBK)	µg/kg		160	81.5 - 229		
5100	Styrene	µg/kg	< 10	0.00		Acceptable *	EPA 8260B
5105	1,1,1,2-Tetrachloroethane	µg/kg		35.4	19.6 - 52.3		
5110	1,1,2,2-Tetrachloroethane	µg/kg		40.8	19.4 - 62.6		
5115	Tetrachloroethylene	µg/kg		138	60.7 - 199		
5140	Toluene	µg/kg		35.1	20.6 - 50.8		
5160	1,1,1-Trichloroethane	µg/kg		101	45.1 - 161		
5165	1,1,2-Trichloroethane	µg/kg		148	98.5 - 198		
5170	Trichloroethylene	µg/kg		90.8	47.6 - 132		
5175	Trichlorofluoromethane	µg/kg		0.00			
5180	1,2,3-Trichloropropane	µg/kg		0.00			
5225	Vinyl acetate	µg/kg		0.00			
5235	Vinyl chloride	µg/kg	< 10	0.00		Acceptable *	EPA 8260B
5260	Total, Xylenes	µg/kg		262	127 - 376		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

*** Indicated result(s) have been revised.**

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-39 Final Complete Report

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Herbicides in Soil							
8545	2,4-D	µg/kg		353	0.00 - 532		
8555	Dalapon	µg/kg	< 100	0.00		Acceptable *	EPA 8151
8560	2,4-DB	µg/kg		255	0.00 - 536		
8595	Dicamba	µg/kg		270	0.00 - 490		
8620	Dinoseb	µg/kg		440	0.00 - 596		
8625	Disulfoton	µg/kg		99.1	0.00 - 189		
7780	MCPP	µg/kg	< 100	0.00		Acceptable *	EPA 8151
6605	Pentachlorophenol	µg/kg		313	0.00 - 416		
8655	2,4,5-T	µg/kg		174	0.00 - 304		
8650	2,4,5-TP (Silvex)	µg/kg		270	0.00 - 383		
PCBs in Soil							
8880	Aroclor 1016	mg/kg	< 0.033	0.00		Acceptable *	EPA 8082
8885	Aroclor 1221	mg/kg	< 0.033	0.00		Acceptable *	EPA 8082
8890	Aroclor 1232	mg/kg	< 0.033	0.00		Acceptable *	EPA 8082
8895	Aroclor 1242	mg/kg	< 0.033	0.00		Acceptable *	EPA 8082
8900	Aroclor 1248	mg/kg	< 0.033	0.00		Acceptable *	EPA 8082
8905	Aroclor 1254	mg/kg	< 0.033	0.00		Acceptable *	EPA 8082
8910	Aroclor 1260	mg/kg		10.3	3.03 - 13.6		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

*** Indicated result(s) have been revised.**

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-39 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 **EPA ID: KS00902** **Report Issued: 10/04/02**

SOIL Study Definitions:

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ERA's Proficiency Testing (PT) Study, Soil-39, has been reviewed by ERA Senior Management and certified compliant with the requirements of the National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program, Chapter 2, June 2000.

The opening date of ERA's Proficiency Testing (PT) Study, Soil-39, was July 29, 2002, and the closing date was September 12, 2002.

Per the requirements of the NELAC Proficiency Testing Program, the Soil-39 results were examined for any study anomalies. A full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the Soil-39 standards met the acceptance criteria contained in the NELAC Proficiency Testing Program, Chapter 2, June 2000.

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's review of the data, no anomalies were noted. All Acceptance Limits for the Soil-39 Study were calculated based on the NELAC Proficiency Testing Program, Chapter 2, June 2000, as applicable.

The Proficiency Testing Study, Soil-39, report shall not be reproduced except in its entirety and without the full written permission of the participating laboratory. The report must not be used by the participating laboratory to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding ERA's Proficiency Testing Study, Soil-39, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

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Study: **SOIL39**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method A**

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-39 Final Complete Report

Tenkasi Viswanathan
QA Director
Analytical Management Labs
Suite B
15130 South Keeler
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 10/04/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil							
1000	Aluminum	mg/kg	3693	5810	1770 - 11300	Acceptable	EPA 6010B
1005	Antimony	mg/kg	189	215	0.00 - 215	Acceptable	EPA 6010B
1010	Arsenic	mg/kg	137	145	93.5 - 181	Acceptable	EPA 6020A
1015	Barium	mg/kg	127	125	96.6 - 185	Acceptable	EPA 6010B
1020	Beryllium	mg/kg	71.3	70.8	48.3 - 86.5	Acceptable	EPA 6010B
1025	Boron	mg/kg	36.7	114	17.5 - 114	Acceptable	EPA 6020A
1030	Cadmium	mg/kg	263	270	171 - 323	Acceptable	EPA 6020A
1035	Calcium	mg/kg	3482	3040	2330 - 4510	Acceptable	EPA 6010B
1040	Chromium	mg/kg	95.5	99.4	67.4 - 125	Acceptable	EPA 6020A
1050	Cobalt	mg/kg	49.3	60.6	31.1 - 60.6	Acceptable	EPA 6010B
1055	Copper	mg/kg	73.2	63.2	43.6 - 90.8	Acceptable	EPA 6010B
1070	Iron	mg/kg	7937	10800	2990 - 20200	Acceptable	EPA 6010B
1075	Lead	mg/kg	80	74.1	54.4 - 96.6	Acceptable	EPA 6020A
1085	Magnesium	mg/kg	1510	1830	1260 - 2760	Acceptable	EPA 6010B
1090	Manganese	mg/kg	249	223	165 - 337	Acceptable	EPA 6010B
1095	Mercury	mg/kg	3.80	3.74	2.11 - 5.97	Acceptable	EPA 6020A
1100	Molybdenum	mg/kg	34.9	32.8	17.3 - 42.7	Acceptable	EPA 6010B
1105	Nickel	mg/kg	80.3	76.3	51.8 - 94.2	Acceptable	EPA 6010B
1125	Potassium	mg/kg	1372	1680	1060 - 2820	Acceptable	EPA 6010B
1140	Selenium	mg/kg	94.6	87.0	49.0 - 113	Acceptable	EPA 6010B
1150	Silver	mg/kg	140	138	63.5 - 193	Acceptable	EPA 6010B
1155	Sodium	mg/kg	372	370	138 - 670	Acceptable	EPA 6010B
1160	Strontium	mg/kg	40.4	41.7	34.5 - 71.3	Acceptable	EPA 6020A
1165	Thallium	mg/kg	133	129	84.2 - 160	Acceptable	EPA 6020A
1175	Tin	mg/kg	55.6	118	31.5 - 118	Acceptable	EPA 6020A
1180	Titanium	mg/kg	113	338	0.00 - 626	Acceptable	EPA 6020A
1185	Vanadium	mg/kg	104	109	67.9 - 144	Acceptable	EPA 6010B
1190	Zinc	mg/kg	141	132	94.7 - 179	Acceptable	EPA 6010B
Cyanide in Soil							
1635	Cyanide, total	mg/kg	64.0	145	0.00 - 236	Acceptable	EPA 9010B
1640	Reactive Cyanide	mg/kg		< 25			
Hexavalent Chromium in Soil							
1045	Chromium VI	mg/kg	66.7	143	14.7 - 157	Acceptable	EPA 7196A

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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SOIL-39 Final Complete Report

Tenkasi Viswanathan
QA Director
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Suite B
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Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 10/04/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil							
5500	Acenaphthene	µg/kg	<	0.00		No Evaluation	EPA 8270C
5505	Acenaphthylene	µg/kg	<	0.00		No Evaluation	EPA 8270C
5545	Aniline	µg/kg		0.00			
5555	Anthracene	µg/kg	1730	3320	733 - 3320	Acceptable	EPA 8270C
5595	Benzidine	µg/kg		0.00			
5610	Benzoic acid	µg/kg		0.00			
5575	Benzo(a)anthracene	µg/kg	787	895	309 - 1140	Acceptable	EPA 8270C
5585	Benzo(b)fluoranthene	µg/kg	5710	7100	1870 - 9670	Acceptable	EPA 8270C
5600	Benzo(k)fluoranthene	µg/kg	2490	3380	790 - 4510	Acceptable	EPA 8270C
5590	Benzo(g,h,i)perylene	µg/kg	<	0.00		No Evaluation	EPA 8270C
5580	Benzo(a)pyrene	µg/kg	1900	3640	782 - 3640	Acceptable	EPA 8270C
5630	Benzyl alcohol	µg/kg		0.00			
5660	4-Bromophenyl-phenylether	µg/kg	6120	7260	2240 - 9620	Acceptable	EPA 8270C
5670	Butylbenzylphthalate	µg/kg	2730	2830	829 - 4050	Acceptable	EPA 8270C
5680	Carbazole	µg/kg	<	0.00		No Evaluation	EPA 8270C
5745	4-Chloroaniline	µg/kg	<	0.00		No Evaluation	EPA 8270C
5760	bis(2-Chloroethoxy)methane	µg/kg	3610	3440	843 - 4140	Acceptable	EPA 8270C
5765	bis(2-Chloroethyl)ether	µg/kg	<	0.00		No Evaluation	EPA 8270C
5780	bis(2-Chloroisopropyl)ether	µg/kg	<	0.00		No Evaluation	EPA 8270C
5700	4-Chloro-3-methylphenol	µg/kg	4350	6990	1230 - 7350	Acceptable	EPA 8270C
5790	1-Chloronaphthalene	µg/kg		0.00			
5795	2-Chloronaphthalene	µg/kg	1200	1420	261 - 1840	Acceptable	EPA 8270C
5800	2-Chlorophenol	µg/kg	5320	8400	750 - 8400	Acceptable	EPA 8270C
5825	4-Chlorophenyl-phenylether	µg/kg	5750	7110	2140 - 9040	Acceptable	EPA 8270C
5855	Chrysene	µg/kg	1130	1220	403 - 1720	Acceptable	EPA 8270C
5895	Dibenz(a,h)anthracene	µg/kg	<	0.00		No Evaluation	EPA 8270C
5905	Dibenzofuran	µg/kg	4370	5930	1780 - 7140	Acceptable	EPA 8270C
5925	Di-n-butylphthalate	µg/kg	6820	7800	1700 - 10800	Acceptable	EPA 8270C
4610	1,2-Dichlorobenzene	µg/kg	<	0.00		No Evaluation	EPA 8270C
4615	1,3-Dichlorobenzene	µg/kg	<	0.00		No Evaluation	EPA 8270C
4620	1,4-Dichlorobenzene	µg/kg	3750	5900	0.00 - 6780	Acceptable	EPA 8270C
5945	3,3'-Dichlorobenzidine	µg/kg	<	0.00		No Evaluation	EPA 8270C
6000	2,4-Dichlorophenol	µg/kg	<	0.00		No Evaluation	EPA 8270C
6005	2,6-Dichlorophenol	µg/kg		0.00			
6070	Diethylphthalate	µg/kg	<	0.00		No Evaluation	EPA 8270C
6130	2,4-Dimethylphenol	µg/kg	<	0.00		No Evaluation	EPA 8270C
6135	Dimethylphthalate	µg/kg	6540	8130	1940 - 10300	Acceptable	EPA 8270C

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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SOIL-39 Final Complete Report

Tenkasi Viswanathan
QA Director
Analytical Management Labs
Suite B
15130 South Keeler
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 10/04/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)							
6140	4,6-Dinitro-2-methylphenol	µg/kg	3090	7780	0.00 - 8170	Acceptable	EPA 8270C
6175	2,4-Dinitrophenol	µg/kg	<	0.00		No Evaluation	EPA 8270C
6185	2,4-Dinitrotoluene	µg/kg	5070	6970	1440 - 8640	Acceptable	EPA 8270C
6190	2,6-Dinitrotoluene	µg/kg	<	0.00		No Evaluation	EPA 8270C
6200	Di-n-octylphthalate	µg/kg	9560	11000	1120 - 17800	Acceptable	EPA 8270C
6255	bis(2-Ethylhexyl)phthalate	µg/kg	4220	4880	920 - 7280	Acceptable	EPA 8270C
6265	Fluoranthene	µg/kg	1260	1410	540 - 1800	Acceptable	EPA 8270C
6270	Fluorene	µg/kg	1200	1430	461 - 1860	Acceptable	EPA 8270C
6275	Hexachlorobenzene	µg/kg	2080	2330	735 - 3050	Acceptable	EPA 8270C
4835	Hexachlorobutadiene	µg/kg	6660	9260	0.00 - 12000	Acceptable	EPA 8270C
6285	Hexachlorocyclopentadiene	µg/kg	<	6840	0.00 - 6840	No Evaluation	EPA 8270C
4840	Hexachloroethane	µg/kg	<	0.00		No Evaluation	EPA 8270C
6315	Indeno(1,2,3-cd)pyrene	µg/kg	<	0.00		No Evaluation	EPA 8270C
6320	Isophorone	µg/kg	5040	4950	957 - 6240	Acceptable	EPA 8270C
6385	2-Methylnaphthalene	µg/kg	<	0.00		No Evaluation	EPA 8270C
6400	2-Methylphenol	µg/kg	1740	7610	0.00 - 7610	Acceptable	EPA 8270C
6412	3 & 4-Methylphenol	µg/kg	<	0.00		No Evaluation	EPA 8270C
5005	Naphthalene	µg/kg	818	1090	137 - 1390	Acceptable	EPA 8270C
6460	2-Nitroaniline	µg/kg	<	0.00		No Evaluation	EPA 8270C
6465	3-Nitroaniline	µg/kg	<	0.00		No Evaluation	EPA 8270C
6470	4-Nitroaniline	µg/kg	<	0.00		No Evaluation	EPA 8270C
5015	Nitrobenzene	µg/kg	5310	7160	610 - 8950	Acceptable	EPA 8270C
6490	2-Nitrophenol	µg/kg	<	0.00		No Evaluation	EPA 8270C
6495	3-Nitrophenol	µg/kg		0.00			
6500	4-Nitrophenol	µg/kg	4840	7420	0.00 - 8770	Acceptable	EPA 8270C
6530	N-Nitrosodimethylamine	µg/kg	<	0.00		No Evaluation	EPA 8270C
6535	N-Nitrosodiphenylamine	µg/kg		0.00			
6545	N-Nitroso-di-n-propylamine	µg/kg	7000	9790	1790 - 12400	Acceptable	EPA 8270C
6605	Pentachlorophenol	µg/kg	5300	8490	1200 - 8490	Acceptable	EPA 8270C
6615	Phenanthrene	µg/kg	955	967	401 - 1340	Acceptable	EPA 8270C
6625	Phenol	µg/kg	9360	13900	1150 - 14800	Acceptable	EPA 8270C
6665	Pyrene	µg/kg	1090	1190	374 - 1650	Acceptable	EPA 8270C
5095	Pyridine	µg/kg		0.00			
5155	1,2,4-Trichlorobenzene	µg/kg	<	0.00		No Evaluation	EPA 8270C
6835	2,4,5-Trichlorophenol	µg/kg	<	0.00		No Evaluation	EPA 8270C
6840	2,4,6-Trichlorophenol	µg/kg	5400	8370	1350 - 8370	Acceptable	EPA 8270C

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-39 Final Complete Report

Tenkasi Viswanathan
QA Director
Analytical Management Labs
Suite B
15130 South Keeler
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 10/04/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil							
5145	2-Amino-1-methylbenzene (o-touluidine)	µg/kg		0.00			
5625	Benzotrichloride	µg/kg		0.00			
5635	Benzyl chloride	µg/kg		0.00			
6350	bis(2-methoxyethyl)phthalate	µg/kg		0.00			
5810	2-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5815	3-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5820	4-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5885	Diamylphthalate	µg/kg		0.00			
5920	2,4-Dibromophenyl-4-nitrophenylether	µg/kg		0.00			
6010	2,4-Dichlorophenyl-3-methyl-4-nitrophenylether	µg/kg		0.00			
6015	2,3-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6020	2,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6025	2,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6030	2,6-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6035	3,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6040	3,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6055	Dicyclohexylphthalate	µg/kg		0.00			
6505	4-Nitrophenylphenylether	µg/kg		0.00			
6195	Dinonylphthalate	µg/kg		0.00			
6280	Hexachlorocyclohexane	µg/kg		0.00			
6305	Hexyl-2-ethylhexylphthalate	µg/kg		0.00			
6335	Maleic anhydride	µg/kg		0.00			
6590	Pentachlorobenzene	µg/kg		0.00			
6595	Pentachlorohexane	µg/kg		0.00			
6717	Tetrachlorobenzenes	µg/kg		0.00			
6705	1,2,3,4-Tetrachlorobenzene	µg/kg		0.00			
6710	1,2,3,5-Tetrachlorobenzene	µg/kg		0.00			
6715	1,2,4,5-Tetrachlorobenzene	µg/kg		0.00			
6730	2,3,4,5-Tetrachlorophenol	µg/kg		0.00			
6735	2,3,4,6-Tetrachlorophenol	µg/kg		0.00			
6740	2,3,5,6-Tetrachlorophenol	µg/kg		0.00			
5150	1,2,3-Trichlorobenzene	µg/kg		0.00			
6800	1,3,5-Trichlorobenzene	µg/kg		0.00			
6845	2,3,4-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6850	2,3,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6855	2,3,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6860	2,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-39 Final Complete Report

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EPA ID: KS00902
ERA Laboratory Code: A5230-01
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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil (Cont.)							
6865	2,4,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6870	3,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
Pesticides in Soil							
7025	Aldrin	µg/kg	223	244	93.1 - 341	Acceptable	EPA 8081
7110	alpha-BHC	µg/kg	134	168	59.9 - 268	Acceptable	EPA 8081
7115	beta-BHC	µg/kg	140	147	10.9 - 213	Acceptable	EPA 8081
7105	delta-BHC	µg/kg	164	189	41.7 - 282	Acceptable	EPA 8081
7120	gamma-BHC(Lindane)	µg/kg	167	137	22.4 - 216	Acceptable	EPA 8081
7240	alpha-Chlordane	µg/kg	<	0.00		No Evaluation	EPA 8270C
7245	gamma-Chlordane	µg/kg	<	0.00		No Evaluation	EPA 8270C
7355	4,4'-DDD	µg/kg	182	212	77.2 - 303	Acceptable	EPA 8081
7360	4,4'-DDE	µg/kg	223	294	97.3 - 427	Acceptable	EPA 8081
7365	4,4'-DDT	µg/kg	147	154	66.2 - 214	Acceptable	EPA 8081
7470	Dieldrin	µg/kg	104	136	60.0 - 186	Acceptable	EPA 8081
7540	Endrin	µg/kg	157	146	74.9 - 211	Acceptable	EPA 8081
7530	Endrin aldehyde	µg/kg	146	270	39.1 - 323	Acceptable	EPA 8081
7535	Endrin ketone	µg/kg	120	204	61.2 - 305	Acceptable	EPA 8081
7510	Endosulfan I	µg/kg	240	411	107 - 541	Acceptable	EPA 8081
7515	Endosulfan II	µg/kg	167	301	70.8 - 379	Acceptable	EPA 8081
7520	Endosulfan sulfate	µg/kg	213	428	152 - 604	Acceptable	EPA 8081
7685	Heptachlor	µg/kg	127	123	51.0 - 171	Acceptable	EPA 8081
7690	Heptachlor epoxide	µg/kg	221	220	92.5 - 354	Acceptable	EPA 8081
7810	Methoxychlor	µg/kg	136	121	30.8 - 195	Acceptable	EPA 8081
Chlordane in Soil							
7250	Chlordane, Technical	µg/kg	319	420	77.4 - 529	Acceptable	EPA 8081A
Toxaphene in Soil							
8250	Toxaphene	µg/kg	247	314	0.00 - 471	Acceptable	EPA 8081A
Volatiles in Soil							
4315	Acetone	µg/kg	191	208	0.00 - 460	Acceptable	EPA 8260B
4320	Acetonitrile	µg/kg		0.00			
4325	Acrolein	µg/kg		0.00			
4375	Benzene	µg/kg	85.9	90.5	55.1 - 130	Acceptable	EPA 8260B
4395	Bromodichloromethane	µg/kg	86.9	93.9	54.2 - 141	Acceptable	EPA 8260B
4400	Bromoform	µg/kg	85.3	94.2	45.5 - 142	Acceptable	EPA 8260B

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)							
4950	Bromomethane	µg/kg	<	0.00		No Evaluation	EPA 8260B
4410	2-Butanone (MEK)	µg/kg	39.8	133	20.5 - 216	Acceptable	EPA 8260B
4450	Carbon disulfide	µg/kg	<	0.00		No Evaluation	EPA 8260B
4455	Carbon tetrachloride	µg/kg	74.2	86.4	33.5 - 140	Acceptable	EPA 8260B
4475	Chlorobenzene	µg/kg	107	120	71.1 - 163	Acceptable	EPA 8260B
4575	Chlorodibromomethane	µg/kg	41.3	45.5	24.6 - 67.8	Acceptable	EPA 8260B
4485	Chloroethane	µg/kg	<	0.00		No Evaluation	EPA 8260B
4500	2-Chloroethylvinylether	µg/kg		0.00			
4505	Chloroform	µg/kg	135	140	84.2 - 202	Acceptable	EPA 8260B
4960	Chloromethane	µg/kg	<	0.00		No Evaluation	EPA 8260B
4570	1,2-Dibromo-3-chloropropane	µg/kg		0.00			
4585	1,2-Dibromoethane (EDB)	µg/kg	76.2	83.0	54.0 - 115	Acceptable	EPA 8260B
4595	Dibromomethane	µg/kg	<	0.00		No Evaluation	EPA 8260B
4610	1,2-Dichlorobenzene	µg/kg	22.9	30.0	11.4 - 44.1	Acceptable	EPA 8260B
4615	1,3-Dichlorobenzene	µg/kg	22.0	30.5	10.1 - 44.7	Acceptable	EPA 8260B
4620	1,4-Dichlorobenzene	µg/kg	81.1	108	37.0 - 155	Acceptable	EPA 8260B
4625	Dichlorodifluoromethane	µg/kg	<	0.00		No Evaluation	EPA 8260B
4630	1,1-Dichloroethane	µg/kg	63.0	66.0	35.1 - 102	Acceptable	EPA 8260B
4635	1,2-Dichloroethane	µg/kg	47.2	48.3	30.9 - 69.3	Acceptable	EPA 8260B
4640	1,1-Dichloroethylene	µg/kg	<	0.00		No Evaluation	EPA 8260B
4645	cis-1,2-Dichloroethylene	µg/kg	<	0.00		No Evaluation	EPA 8260B
4700	trans-1,2-Dichloroethylene	µg/kg	<	0.00		No Evaluation	EPA 8260B
4655	1,2-Dichloropropane	µg/kg	<	0.00		No Evaluation	EPA 8260B
4680	cis-1,3-Dichloropropylene	µg/kg	<	0.00		No Evaluation	EPA 8260B
4685	trans-1,3-Dichloropropylene	µg/kg	<	0.00		No Evaluation	EPA 8260B
4765	Ethylbenzene	µg/kg	99.6	110	63.2 - 157	Acceptable	EPA 8260B
4860	2-Hexanone	µg/kg	<	0.00		No Evaluation	EPA 8260B
4975	Methylene chloride	µg/kg	95.9	101	46.2 - 152	Acceptable	EPA 8260B
4995	4-Methyl-2-pentanone (MIBK)	µg/kg	154	160	81.5 - 229	Acceptable	EPA 8260B
5100	Styrene	µg/kg	<	0.00		No Evaluation	EPA 8260B
5105	1,1,1,2-Tetrachloroethane	µg/kg	33.6	35.4	19.6 - 52.3	Acceptable	EPA 8260B
5110	1,1,2,2-Tetrachloroethane	µg/kg	37.6	40.8	19.4 - 62.6	Acceptable	EPA 8260B
5115	Tetrachloroethylene	µg/kg	122	138	60.7 - 199	Acceptable	EPA 8260B
5140	Toluene	µg/kg	34.0	35.1	20.6 - 50.8	Acceptable	EPA 8260B
5160	1,1,1-Trichloroethane	µg/kg	90.5	101	45.1 - 161	Acceptable	EPA 8260B
5165	1,1,2-Trichloroethane	µg/kg	139	148	98.5 - 198	Acceptable	EPA 8260B
5170	Trichloroethylene	µg/kg	83.0	90.8	47.6 - 132	Acceptable	EPA 8260B

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-39 Final Complete Report

Tenkasi Viswanathan
QA Director
Analytical Management Labs
Suite B
15130 South Keeler
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 10/04/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)							
5175	Trichlorofluoromethane	µg/kg		0.00			
5180	1,2,3-Trichloropropane	µg/kg		0.00			
5225	Vinyl acetate	µg/kg		0.00			
5235	Vinyl chloride	µg/kg	<	0.00		No Evaluation	EPA 8260B
5260	Total, Xylenes	µg/kg	235	262	127 - 376	Acceptable	EPA 8260B
Herbicides in Soil							
8545	2,4-D	µg/kg	277	353	0.00 - 532	Acceptable	EPA 8151
8555	Dalapon	µg/kg	<	0.00		No Evaluation	EPA 8151
8560	2,4-DB	µg/kg	258	255	0.00 - 536	Acceptable	EPA 8151
8595	Dicamba	µg/kg	202	270	0.00 - 490	Acceptable	EPA 8151
8620	Dinoseb	µg/kg	221	440	0.00 - 596	Acceptable	EPA 8151
8625	Disulfoton	µg/kg		99.1	0.00 - 189		
7780	MCPP	µg/kg	<	0.00		No Evaluation	EPA 8151
6605	Pentachlorophenol	µg/kg		313	0.00 - 416		
8655	2,4,5-T	µg/kg	120	174	0.00 - 304	Acceptable	EPA 8151
8650	2,4,5-TP (Silvex)	µg/kg	217	270	0.00 - 383	Acceptable	EPA 8151
PCBs in Soil							
8880	Aroclor 1016	mg/kg	<	0.00		No Evaluation	EPA 8082
8885	Aroclor 1221	mg/kg	<	0.00		No Evaluation	EPA 8082
8890	Aroclor 1232	mg/kg	<	0.00		No Evaluation	EPA 8082
8895	Aroclor 1242	mg/kg	<	0.00		No Evaluation	EPA 8082
8900	Aroclor 1248	mg/kg	<	0.00		No Evaluation	EPA 8082
8905	Aroclor 1254	mg/kg	<	0.00		No Evaluation	EPA 8082
8910	Aroclor 1260	mg/kg	8.56	10.3	3.03 - 13.6	Acceptable	EPA 8082

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-42 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 **EPA ID: KS00902**

Report Issued: 08/01/03
Study Dates: 04/28/03 - 06/12/03

SOIL Study Definitions:

The reported value is the value that the laboratory reported to ERA.

The ERA assigned value for the Organic Proficiency Testing Standards is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation as applicable. The ERA assigned value for the Inorganic Proficiency Testing Standards is equal to the maximum amount of the parameter available in the standard by applicable EPA methodologies. All NELAC parameters not added to a standard are given an assigned Value of "0", per the guidance issued by the NELAC Board of Directors, on December 14, 2000. Non-NELAC parameters not added to a standard may be given an assigned value of less than a minimum verified concentration as determined in the background soil for applicable EPA methodologies.

The acceptance limits are established per the NELAC PT program criteria or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For a further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside of the Acceptance Limits.
No Evaluation	=	Reported Value cannot be evaluated.

The method description is the method the laboratory reported to ERA.

SOIL Study Discussion:

ERA's Proficiency Testing (PT) Study, SOIL-42, has been reviewed by ERA Senior Management and certified compliant with the requirements of the National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program Standards, Chapter 2, June 2000.

Per the requirements of the NELAC Proficiency Testing Program, a full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the SOIL-42 standards met the acceptance criteria contained in the NELAC Proficiency Testing Program Standards, Chapter 2, June 2000. If the analyte is included in the NELAC Fields of Testing list the acceptance limits were calculated based on the NELAC Proficiency Testing Program Standards, Chapter 2, June 2000. If the analyte is not included in the NELAC Fields of Testing list, the acceptance limits were calculated using the procedures outlined in ERA's Standard Operating Procedure for the Generation of Performance Acceptance Limits (SOP 0260, Rev. 2.0).

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's review of the data one anomaly was noted. This anomaly is noted on the following page.

The Proficiency Testing Study, SOIL-42, report shall not be reproduced except in its entirety and not without the permission of the participating laboratory. The report must not be used by the participating laboratory to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding ERA's Proficiency Testing Study, SOIL-42, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

ERA Laboratory Code: A5230-01 **EPA ID: KS00902**

Report Issued: 08/01/03
Study Dates: 04/28/03 - 06/12/03

Study Discussion: Soil-42 - PCBs in Soil

During the review of the Soil-42 PCBs in Soil data set ERA discovered a failure rate of 24.2% for Aroclor 1232 and 18.9% for Aroclor 1242. As we believe these failure rates are high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification and stability data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'.

Upon further examination of the PCBs in Soil data set ERA found that sixteen labs had misidentified Aroclor 1232 as Aroclor 1242. If you are one of the laboratories that misidentified Aroclor 1232 and would like technical assistance please call ERA's Organic Chemistry Department at 1-800-372-0122.

NVLAP® Lab code: 200388-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

Study: **SOIL-42**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method A**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200388-0

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-42 Final Complete Report

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Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 08/01/03
Study Dates: 04/28/03 - 06/12/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil							
1000	Aluminum	mg/kg	3550	6180	2280 - 10900	Acceptable	EPA 6010B
1005	Antimony	mg/kg	128	177	0.00 - 177	Acceptable	EPA 6010B
1010	Arsenic	mg/kg	248	327	202 - 394	Acceptable	EPA 6020A
1015	Barium	mg/kg	240	292	215 - 381	Acceptable	EPA 6010B
1020	Beryllium	mg/kg	88.5	111	72.4 - 121	Acceptable	EPA 6010B
1025	Boron	mg/kg	132	174	82.2 - 174	Acceptable	EPA 6020A
1030	Cadmium	mg/kg	94	96.1	70.7 - 118	Acceptable	EPA 6020A
1035	Calcium	mg/kg	3020	3180	2410 - 4090	Acceptable	EPA 6010B
1040	Chromium	mg/kg	82	115	76.6 - 135	Acceptable	EPA 6020A
1050	Cobalt	mg/kg	97	126	72.3 - 126	Acceptable	EPA 6010B
1055	Copper	mg/kg	98	112	79.5 - 135	Acceptable	EPA 6010B
1070	Iron	mg/kg	7430	10800	2840 - 19800	Acceptable	EPA 6010B
1075	Lead	mg/kg	91	109	73.6 - 138	Acceptable	EPA 6020A
1085	Magnesium	mg/kg	1810	2360	1580 - 3240	Acceptable	EPA 6010B
1090	Manganese	mg/kg	215	234	169 - 315	Acceptable	EPA 6010B
1095	Mercury	mg/kg	9.50	11.8	4.18 - 17.6	Acceptable	EPA 6020A
1100	Molybdenum	mg/kg	48	58.1	34.1 - 68.3	Acceptable	EPA 6010B
1105	Nickel	mg/kg	101	116	75.4 - 121	Acceptable	EPA 6010B
1125	Potassium	mg/kg	1490	2090	1350 - 3070	Acceptable	EPA 6010B
1140	Selenium	mg/kg	80	90.5	54.1 - 112	Acceptable	EPA 6010B
1150	Silver	mg/kg	43	102	57.5 - 130	Not Acceptable	EPA 6010B
1155	Sodium	mg/kg	510	620	331 - 829	Acceptable	EPA 6010B
1160	Strontium	mg/kg	79.6	90.8	63.0 - 121	Acceptable	EPA 6020A
1165	Thallium	mg/kg	93.6	115	73.2 - 133	Acceptable	EPA 6020A
1175	Tin	mg/kg	38.3	119	29.1 - 119	Acceptable	EPA 6020A
1180	Titanium	mg/kg	122	338	0.00 - 633	Acceptable	EPA 6020A
1185	Vanadium	mg/kg	96	125	81.9 - 152	Acceptable	EPA 6010B
1190	Zinc	mg/kg	338	391	245 - 437	Acceptable	EPA 6010B
Cyanide in Soil							
1635	Cyanide, total	mg/kg	202	235	0.00 - 376	Acceptable	EPA 9014
1640	Reactive Cyanide	mg/kg		<25.0	<25.0		
Hexavalent Chromium in Soil							
1045	Chromium VI	mg/kg	120	165	53.1 - 181	Acceptable	EPA 7196A

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-42 Final Complete Report

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Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 08/01/03
Study Dates: 04/28/03 - 06/12/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil							
5500	Acenaphthene	µg/kg	3150	5670	1580 - 6710	Acceptable	EPA 8270C
5505	Acenaphthylene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5545	Aniline	µg/kg		0.00			
5555	Anthracene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5595	Benzidine	µg/kg	< 330	0.00		Acceptable	EPA 8270C
5610	Benzoic acid	µg/kg	< 330	0.00		Acceptable	EPA 8270C
5575	Benzo(a)anthracene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5585	Benzo(b)fluoranthene	µg/kg	2910	4590	1290 - 6030	Acceptable	EPA 8270C
5600	Benzo(k)fluoranthene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5590	Benzo(g,h,i)perylene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5580	Benzo(a)pyrene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5630	Benzyl alcohol	µg/kg	< 330	0.00		Acceptable	EPA 8270C
5660	4-Bromophenyl-phenylether	µg/kg	5900	10800	3120 - 13900	Acceptable	EPA 8270C
5670	Butylbenzylphthalate	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5680	Carbazole	µg/kg	< 330	0.00		Acceptable	EPA 8270C
5745	4-Chloroaniline	µg/kg	< 330	0.00		Acceptable	EPA 8270C
5760	bis(2-Chloroethoxy)methane	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5765	bis(2-Chloroethyl)ether	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5780	bis(2-Chloroisopropyl)ether	µg/kg	< 125	1750	386 - 2050	Not Acceptable	EPA 8270C
5700	4-Chloro-3-methylphenol	µg/kg	3040	7700	1620 - 7700	Acceptable	EPA 8270C
5790	1-Chloronaphthalene	µg/kg		0.00			
5795	2-Chloronaphthalene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5800	2-Chlorophenol	µg/kg	2840	8840	1410 - 8840	Acceptable	EPA 8270C
5825	4-Chlorophenyl-phenylether	µg/kg	3580	5770	1830 - 7130	Acceptable	EPA 8270C
5855	Chrysene	µg/kg	2397	3950	1100 - 5260	Acceptable	EPA 8270C
5895	Dibenz(a,h)anthracene	µg/kg	644	839	138 - 1210	Acceptable	EPA 8270C
5905	Dibenzofuran	µg/kg	5130	8680	2120 - 10800	Acceptable	EPA 8270C
5925	Di-n-butylphthalate	µg/kg	5150	10500	2110 - 13500	Acceptable	EPA 8270C
4610	1,2-Dichlorobenzene	µg/kg	< 330	0.00		Acceptable	EPA 8270C
4615	1,3-Dichlorobenzene	µg/kg	< 330	0.00		Acceptable	EPA 8270C
4620	1,4-Dichlorobenzene	µg/kg	< 330	0.00		Acceptable	EPA 8270C
5945	3,3'-Dichlorobenzidine	µg/kg	< 330	0.00		Acceptable	EPA 8270C
6000	2,4-Dichlorophenol	µg/kg	3950	11100	1350 - 11100	Acceptable	EPA 8270C
6005	2,6-Dichlorophenol	µg/kg		0.00			
6070	Diethylphthalate	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6130	2,4-Dimethylphenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6135	Dimethylphthalate	µg/kg	1790	5800	1120 - 7240	Acceptable	EPA 8270C

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-42 Final Complete Report

Tenkasi S. Viswanathan
QC Manager
Analytical Management Labs
Suite B
15130 S Keeler
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 08/01/03
Study Dates: 04/28/03 - 06/12/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)							
6360	4,6-Dinitro-2-methylphenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6175	2,4-Dinitrophenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6185	2,4-Dinitrotoluene	µg/kg	4200	8270	1100 - 10200	Acceptable	EPA 8270C
6190	2,6-Dinitrotoluene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6200	Di-n-octylphthalate	µg/kg	2280	3460	833 - 4790	Acceptable	EPA 8270C
6255	bis(2-Ethylhexyl)phthalate	µg/kg	2830	4380	915 - 5990	Acceptable	EPA 8270C
6265	Fluoranthene	µg/kg	5160	8170	2380 - 10300	Acceptable	EPA 8270C
6270	Fluorene	µg/kg	2310	3480	1060 - 4280	Acceptable	EPA 8270C
6275	Hexachlorobenzene	µg/kg	4110	6940	1980 - 8700	Acceptable	EPA 8270C
4835	Hexachlorobutadiene	µg/kg	2250	4480	495 - 5450	Acceptable	EPA 8270C
6285	Hexachlorocyclopentadiene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
4840	Hexachloroethane	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6315	Indeno(1,2,3-cd)pyrene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6320	Isophorone	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6385	2-Methylnaphthalene	µg/kg	< 330	0.00		Acceptable	EPA 8270C
6400	2-Methylphenol	µg/kg	717	5610	54.0 - 5610	Acceptable	EPA 8270C
6412	3 & 4-Methylphenol	µg/kg	< 330	0.00		Acceptable	EPA 8270C
5005	Naphthalene	µg/kg	2700	5250	925 - 6060	Acceptable	EPA 8270C
6460	2-Nitroaniline	µg/kg	< 330	0.00		Acceptable	EPA 8270C
6465	3-Nitroaniline	µg/kg	< 330	0.00		Acceptable	EPA 8270C
6470	4-Nitroaniline	µg/kg	< 330	0.00		Acceptable	EPA 8270C
5015	Nitrobenzene	µg/kg	982	1940	385 - 2280	Acceptable	EPA 8270C
6490	2-Nitrophenol	µg/kg	2500	6910	710 - 7130	Acceptable	EPA 8270C
6495	3-Nitrophenol	µg/kg		0.00			
6500	4-Nitrophenol	µg/kg	1660	7540	0.00 - 9600	Acceptable	EPA 8270C
6530	N-Nitrosodimethylamine	µg/kg	< 330	0.00		Acceptable	EPA 8270C
6535	N-Nitrosodiphenylamine	µg/kg	< 330	0.00		Acceptable	EPA 8270C
6545	N-Nitroso-di-n-propylamine	µg/kg	1940	5940	630 - 7470	Acceptable	EPA 8270C
6605	Pentachlorophenol	µg/kg	2250	7460	120 - 7680	Acceptable	EPA 8270C
6615	Phenanthrene	µg/kg	3830	5740	1930 - 7330	Acceptable	EPA 8270C
6625	Phenol	µg/kg	784	2420	309 - 2510	Acceptable	EPA 8270C
6665	Pyrene	µg/kg	3930	6870	1470 - 9330	Acceptable	EPA 8270C
5095	Pyridine	µg/kg		0.00			
5155	1,2,4-Trichlorobenzene	µg/kg	4990	11300	1550 - 13100	Acceptable	EPA 8270C
6835	2,4,5-Trichlorophenol	µg/kg	2000	5020	487 - 5350	Acceptable	EPA 8270C
6840	2,4,6-Trichlorophenol	µg/kg	3430	9200	1320 - 9300	Acceptable	EPA 8270C

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-42 Final Complete Report

Tenkasi S. Viswanathan
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Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 08/01/03
Study Dates: 04/28/03 - 06/12/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil							
5145	2-Amino-1-methylbenzene (o-toluidine)	µg/kg		0.00			
5625	Benzotrichloride	µg/kg		0.00			
5635	Benzyl chloride	µg/kg		0.00			
6350	bis(2-methoxyethyl)phthalate	µg/kg		0.00			
5810	2-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5815	3-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5820	4-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5885	Diamylphthalate	µg/kg		0.00			
5920	2,4-Dibromophenyl-4-nitrophenylether	µg/kg		0.00			
6010	2,4-Dichlorophenyl-3-methyl-4-nitrophenylether	µg/kg		0.00			
6015	2,3-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6020	2,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6025	2,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6030	2,6-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6035	3,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6040	3,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6055	Dicyclohexylphthalate	µg/kg		0.00			
6505	4-Nitrophenylphenylether	µg/kg		0.00			
6195	Dinonylphthalate	µg/kg		0.00			
6280	Hexachlorocyclohexane	µg/kg		0.00			
6305	Hexyl-2-ethylhexylphthalate	µg/kg		0.00			
6335	Maleic anhydride	µg/kg		0.00			
6590	Pentachlorobenzene	µg/kg		0.00			
6595	Pentachlorohexane	µg/kg		0.00			
6717	Tetrachlorobenzenes	µg/kg		0.00			
6705	1,2,3,4-Tetrachlorobenzene	µg/kg		0.00			
6710	1,2,3,5-Tetrachlorobenzene	µg/kg		0.00			
6715	1,2,4,5-Tetrachlorobenzene	µg/kg		0.00			
6730	2,3,4,5-Tetrachlorophenol	µg/kg		0.00			
6735	2,3,4,6-Tetrachlorophenol	µg/kg		0.00			
6740	2,3,5,6-Tetrachlorophenol	µg/kg		0.00			
5150	1,2,3-Trichlorobenzene	µg/kg		0.00			
6800	1,3,5-Trichlorobenzene	µg/kg		0.00			
6845	2,3,4-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6850	2,3,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6855	2,3,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6860	2,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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SOIL-42 Final Complete Report

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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 08/01/03
Study Dates: 04/28/03 - 06/12/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil (Cont.)							
6865	2,4,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6870	3,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
Pesticides in Soil							
7025	Aldrin	µg/kg	190	259	93.1 - 359	Acceptable	EPA 8081A
7110	alpha-BHC	µg/kg	151	199	57.6 - 284	Acceptable	EPA 8081A
7115	beta-BHC	µg/kg	43	127	8.40 - 214	Acceptable	EPA 8081A
7105	delta-BHC	µg/kg	63	184	53.2 - 267	Acceptable	EPA 8081A
7120	gamma-BHC(Lindane)	µg/kg	98	133	28.0 - 214	Acceptable	EPA 8081A
7240	alpha-Chlordane	µg/kg	< 3.3	0.00		Acceptable	EPA 8081A
7245	gamma-Chlordane	µg/kg	< 3.3	0.00		Acceptable	EPA 8081A
7355	4,4'-DDD	µg/kg	160	270	105 - 385	Acceptable	EPA 8081A
7360	4,4'-DDE	µg/kg	271	466	166 - 661	Acceptable	EPA 8081A
7365	4,4'-DDT	µg/kg	94	201	46.5 - 296	Acceptable	EPA 8081A
7470	Dieldrin	µg/kg	159	236	101 - 311	Acceptable	EPA 8081A
7540	Endrin	µg/kg	71	117	34.2 - 182	Acceptable	EPA 8081A
7530	Endrin aldehyde	µg/kg	22.9	267	0.00 - 273	Acceptable	EPA 8081A
7535	Endrin ketone	µg/kg	34.8	211	65.3 - 293	Not Acceptable	EPA 8081A
7510	Endosulfan I	µg/kg	113	261	59.7 - 261	Acceptable	EPA 8081A
7515	Endosulfan II	µg/kg	9.69	196	59.0 - 245	Not Acceptable	EPA 8081A
7520	Endosulfan sulfate	µg/kg	10.6	268	53.7 - 402	Not Acceptable	EPA 8081A
7685	Heptachlor	µg/kg	253	377	131 - 505	Acceptable	EPA 8081A
7690	Heptachlor epoxide	µg/kg	166	245	89.8 - 332	Acceptable	EPA 8081A
7810	Methoxychlor	µg/kg	< 1830	0.00		Acceptable	EPA 8081A
Chlordane in Soil							
7250	Chlordane, Technical	µg/kg	205	259	70.0 - 334	Acceptable	EPA 8081A
Toxaphene in Soil							
8250	Toxaphene	µg/kg	383	381	0.00 - 554	Acceptable	EPA 8081A
Volatiles in Soil							
4315	Acetone	µg/kg	153	173	0.00 - 322	Acceptable	EPA 8260B
4320	Acetonitrile	µg/kg	< 20	0.00		Acceptable	EPA 8260B
4325	Acrolein	µg/kg	< 20	0.00		Acceptable	EPA 8260B
4375	Benzene	µg/kg	104	118	66.1 - 158	Acceptable	EPA 8260B
4395	Bromodichloromethane	µg/kg	112	123	76.2 - 170	Acceptable	EPA 8260B
4400	Bromoform	µg/kg	< 10	0.00		Acceptable	EPA 8260B

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

Tenkasi S. Viswanathan
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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 08/01/03
Study Dates: 04/28/03 - 06/12/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)							
4950	Bromomethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4410	2-Butanone (MEK)	µg/kg	32	103	0.00 - 160	Acceptable	EPA 8260B
4450	Carbon disulfide	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4455	Carbon tetrachloride	µg/kg	31	34.6	19.4 - 50.6	Acceptable	EPA 8260B
4475	Chlorobenzene	µg/kg	142	158	95.6 - 208	Acceptable	EPA 8260B
4575	Chlorodibromomethane	µg/kg	39	42.8	24.9 - 60.1	Acceptable	EPA 8260B
4485	Chloroethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4500	2-Chloroethylvinylether	µg/kg		0.00			
4505	Chloroform	µg/kg	143	156	103 - 207	Acceptable	EPA 8260B
4960	Chloromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4570	1,2-Dibromo-3-chloropropane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4585	1,2-Dibromoethane (EDB)	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4595	Dibromomethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4610	1,2-Dichlorobenzene	µg/kg	53	66.0	17.1 - 102	Acceptable	EPA 8260B
4615	1,3-Dichlorobenzene	µg/kg	86	146	38.9 - 223	Acceptable	EPA 8260B
4620	1,4-Dichlorobenzene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4625	Dichlorodifluoromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4630	1,1-Dichloroethane	µg/kg	56	58.0	33.8 - 82.0	Acceptable	EPA 8260B
4635	1,2-Dichloroethane	µg/kg	87	94.7	56.3 - 134	Acceptable	EPA 8260B
4640	1,1-Dichloroethylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4645	cis-1,2-Dichloroethylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4700	trans-1,2-Dichloroethylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4655	1,2-Dichloropropane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4680	cis-1,3-Dichloropropylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4685	trans-1,3-Dichloropropylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4765	Ethylbenzene	µg/kg	163	177	96.1 - 248	Acceptable	EPA 8260B
4860	2-Hexanone	µg/kg	< 10	< 10.0		Acceptable	EPA 8260B
4975	Methylene chloride	µg/kg	151	161	76.9 - 231	Acceptable	EPA 8260B
4995	4-Methyl-2-pentanone (MIBK)	µg/kg	100	94.1	36.2 - 144	Acceptable	EPA 8260B
5100	Styrene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5105	1,1,1,2-Tetrachloroethane	µg/kg	148	172	100 - 232	Acceptable	EPA 8260B
5110	1,1,2,2-Tetrachloroethane	µg/kg	153	175	91.8 - 250	Acceptable	EPA 8260B
5115	Tetrachloroethylene	µg/kg	32	30.5	15.1 - 47.5	Acceptable	EPA 8260B
5140	Toluene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5160	1,1,1-Trichloroethane	µg/kg	106	121	72.1 - 164	Acceptable	EPA 8260B
5165	1,1,2-Trichloroethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5170	Trichloroethylene	µg/kg	89	84.9	51.8 - 122	Acceptable	EPA 8260B

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-42 Final Complete Report

Tenkasi S. Viswanathan
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Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 08/01/03
Study Dates: 04/28/03 - 06/12/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)							
5175	Trichlorofluoromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5180	1,2,3-Trichloropropane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5225	Vinyl acetate	µg/kg	< 20	0.00		Acceptable	EPA 8260B
5235	Vinyl chloride	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5260	Total, Xylenes	µg/kg	185	166	85.5 - 245	Acceptable	EPA 8260B
Herbicides in Soil							
8545	2,4-D	µg/kg	274	481	0.00 - 706	Acceptable	EPA 8151A
8555	Dalapon	µg/kg		0.00			
8560	2,4-DB	µg/kg		749	0.00 - 1180		
8595	Dicamba	µg/kg	46	57.3	0.00 - 112	Acceptable	EPA 8151A
8620	Dinoseb	µg/kg		298	0.00 - 506		
7780	MCPP	µg/kg		0.00			
6605	Pentachlorophenol	µg/kg		962	0.00 - 1310		
8655	2,4,5-T	µg/kg	129	268	0.00 - 441	Acceptable	EPA 8151A
8650	2,4,5-TP (Silvex)	µg/kg	92	108	0.00 - 193	Acceptable	EPA 8151A
PCBs in Soil							
8880	Aroclor 1016	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8885	Aroclor 1221	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8890	Aroclor 1232	mg/kg	< 0.05	1.60	0.366 - 2.27	Not Acceptable	EPA 8082A
8895	Aroclor 1242	mg/kg	0.610	0.00		Not Acceptable	EPA 8082A
8900	Aroclor 1248	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8905	Aroclor 1254	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8910	Aroclor 1260	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

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SOIL-44 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 **EPA ID: KS00902**

Report Issued: 12/30/03
Study Dates: 10/24/03 - 12/08/03

SOIL Study Definitions:

The reported value is the value that the laboratory reported to ERA.

The ERA assigned value for the Organic Proficiency Testing Standards is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation as applicable. The ERA assigned value for the Inorganic Proficiency Testing Standards is equal to the maximum amount of the parameter available in the standard by applicable EPA methodologies. All NELAC parameters not added to a standard are given an assigned Value of "0", per the guidance issued by the NELAC Board of Directors, on December 14, 2000. Non-NELAC parameters not added to a standard may be given an assigned value of less than a minimum verified concentration as determined in the background soil for applicable EPA methodologies.

The acceptance limits are established per the NELAC PT program criteria or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For a further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside of the Acceptance Limits.
No Evaluation	=	Reported Value cannot be evaluated.

The method description is the method the laboratory reported to ERA.

SOIL Study Discussion:

ERA's Proficiency Testing (PT) Study, SOIL-44, has been reviewed by ERA Senior Management and certified compliant with the requirements of the National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program Standards, Chapter 2, July 2003.

Per the requirements of the NELAC Proficiency Testing Program, a full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the SOIL-44 standards met the acceptance criteria contained in the NELAC Proficiency Testing Program Standards, Chapter 2, July 2003. If the analyte is included in the NELAC Fields of Testing list, the acceptance limits were calculated based on the NELAC Proficiency Testing Program Standards, Chapter 2, July 2003. If the analyte is not included in the NELAC Fields of Testing list, the acceptance limits were calculated using the procedures outlined in ERA's Standard Operating Procedure for the Generation of Performance Acceptance Limits (SOP 0260, Rev. 2.0).

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's review of the data no anomalies were noted.

The Proficiency Testing Study, SOIL-44, report shall not be reproduced except in its entirety and not without the permission of the participating laboratory. The report must not be used by the participating laboratory to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding ERA's Proficiency Testing Study, SOIL-44, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

ENVIRONMENTAL
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Study: **SOIL-44**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Laboratories**

Report Type: **Complete**

Report Method: **Method A**

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-44 Final Complete Report

Tenkasi S Viswanathan
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Analytical Management Laboratories
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Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/30/03
Study Dates: 10/24/03 - 12/08/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil							
1000	Aluminum	mg/kg	5400	7260	3880 - 11400	Acceptable	EPA 6010B
1005	Antimony	mg/kg	197	223	0.00 - 223	Acceptable	EPA 6010B
1010	Arsenic	mg/kg	104	101	72.7 - 119	Acceptable	EPA 6010B
1015	Barium	mg/kg	149	145	121 - 193	Acceptable	EPA 6010B
1020	Beryllium	mg/kg	51.3	54.4	39.8 - 61.2	Acceptable	EPA 6010B
1025	Boron	mg/kg		144	44.4 - 144		
1030	Cadmium	mg/kg	59.8	64.4	42.5 - 66.5	Acceptable	EPA 6010B
1035	Calcium	mg/kg	4070	3490	2950 - 4650	Acceptable	EPA 6010B
1040	Chromium	mg/kg	69.2	71.9	50.5 - 85.5	Acceptable	EPA 6010B
1050	Cobalt	mg/kg	84.1	98.1	58.8 - 98.1	Acceptable	EPA 6010B
1055	Copper	mg/kg	87.2	81.7	65.1 - 101	Acceptable	EPA 6010B
1070	Iron	mg/kg	9620	10800	3960 - 19400	Acceptable	EPA 6010B
1075	Lead	mg/kg	130	126	93.5 - 150	Acceptable	EPA 6010B
1085	Magnesium	mg/kg	2420	2600	1960 - 3460	Acceptable	EPA 6010B
1090	Manganese	mg/kg	491	465	361 - 571	Acceptable	EPA 6010B
1095	Mercury	mg/kg	4.02	5.82	2.74 - 7.26	Acceptable	EPA 7471A
1100	Molybdenum	mg/kg	27.5	25.9	15.4 - 31.2	Acceptable	EPA 6010B
1105	Nickel	mg/kg	55.5	58.3	41.3 - 66.7	Acceptable	EPA 6010B
1125	Potassium	mg/kg	1740	1980	1470 - 2880	Acceptable	EPA 6010B
1140	Selenium	mg/kg	119	111	73.7 - 132	Acceptable	EPA 6010B
1150	Silver	mg/kg	98.8	114	75.4 - 135	Acceptable	EPA 6010B
1155	Sodium	mg/kg	203	206	97.1 - 375	Acceptable	EPA 6010B
1160	Strontium	mg/kg		41.7	34.7 - 69.7		
1165	Thallium	mg/kg	191	199	135 - 229	Acceptable	EPA 6010B
1175	Tin	mg/kg		164	68.4 - 164		
1180	Titanium	mg/kg		338	0.00 - 661		
1185	Vanadium	mg/kg	117	122	84.2 - 148	Acceptable	EPA 6010B
1190	Zinc	mg/kg	255	247	181 - 303	Acceptable	EPA 6010B
Cyanide in Soil							
1635	Cyanide, total	mg/kg	93.9	103	0.00 - 147	Acceptable	EPA 9010B
1640	Reactive Cyanide	mg/kg		< 25.0	< 25.0		
Hexavalent Chromium in Soil							
1045	Chromium VI	mg/kg	44.5	137	4.40 - 137	Acceptable	EPA 7196A

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-44 Final Complete Report

Tenkasi S Viswanathan
QA Director
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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/30/03
Study Dates: 10/24/03 - 12/08/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil							
5500	Acenaphthene	µg/kg	1850	2480	773 - 2870	Acceptable	EPA 8270C
5505	Acenaphthylene	µg/kg	2490	3570	994 - 3570	Acceptable	EPA 8270C
5545	Aniline	µg/kg		0.00			
5555	Anthracene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5595	Benzidine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5610	Benzoic acid	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5575	Benzo(a)anthracene	µg/kg	1130	1480	584 - 1740	Acceptable	EPA 8270C
5585	Benzo(b)fluoranthene	µg/kg	864	1050	258 - 1360	Acceptable	EPA 8270C
5600	Benzo(k)fluoranthene	µg/kg	789	1000	315 - 1340	Acceptable	EPA 8270C
5590	Benzo(g,h,i)perylene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5580	Benzo(a)pyrene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5630	Benzyl alcohol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5660	4-Bromophenyl-phenylether	µg/kg	3300	4430	1530 - 5470	Acceptable	EPA 8270C
5670	Butylbenzylphthalate	µg/kg	4670	6210	1230 - 8790	Acceptable	EPA 8270C
5680	Carbazole	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5745	4-Chloroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5760	bis(2-Chloroethoxy)methane	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5765	bis(2-Chloroethyl)ether	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5780	bis(2-Chloroisopropyl)ether	µg/kg	2260	7090	790 - 8710	Acceptable	EPA 8270C
5700	4-Chloro-3-methylphenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5790	1-Chloronaphthalene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5795	2-Chloronaphthalene	µg/kg	1770	2330	606 - 2750	Acceptable	EPA 8270C
5800	2-Chlorophenol	µg/kg	2550	5090	263 - 5240	Acceptable	EPA 8270C
5825	4-Chlorophenyl-phenylether	µg/kg	6640	8910	2630 - 11000	Acceptable	EPA 8270C
5855	Chrysene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5895	Dibenz(a,h)anthracene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5905	Dibenzofuran	µg/kg	4410	6960	1650 - 8670	Acceptable	EPA 8270C
5925	Di-n-butylphthalate	µg/kg	4880	8270	1680 - 11300	Acceptable	EPA 8270C
4610	1,2-Dichlorobenzene	µg/kg	2770	4370	557 - 4700	Acceptable	EPA 8270C
4615	1,3-Dichlorobenzene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
4620	1,4-Dichlorobenzene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5945	3,3'-Dichlorobenzidine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6000	2,4-Dichlorophenol	µg/kg	3950	8230	530 - 8750	Acceptable	EPA 8270C
6005	2,6-Dichlorophenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6070	Diethylphthalate	µg/kg	3060	4210	638 - 5700	Acceptable	EPA 8270C
6130	2,4-Dimethylphenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6135	Dimethylphthalate	µg/kg	< 125	0.00		Acceptable	EPA 8270C

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-44 Final Complete Report

Tenkasi S Viswanathan
QA Director
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Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/30/03
Study Dates: 10/24/03 - 12/08/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)							
6360	4,6-Dinitro-2-methylphenol	µg/kg	2330	7720	0.00 - 8120	Acceptable	EPA 8270C
6175	2,4-Dinitrophenol	µg/kg	182	6390	0.00 - 6390	Acceptable	EPA 8270C
6185	2,4-Dinitrotoluene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6190	2,6-Dinitrotoluene	µg/kg	3680	4650	1240 - 5660	Acceptable	EPA 8270C
6200	Di-n-octylphthalate	µg/kg	2790	3740	695 - 5350	Acceptable	EPA 8270C
6255	bis(2-Ethylhexyl)phthalate	µg/kg	5290	8100	1190 - 11700	Acceptable	EPA 8270C
6265	Fluoranthene	µg/kg	3250	4640	1550 - 5730	Acceptable	EPA 8270C
6270	Fluorene	µg/kg	2250	3060	898 - 3680	Acceptable	EPA 8270C
6275	Hexachlorobenzene	µg/kg	4140	5790	1810 - 7110	Acceptable	EPA 8270C
4835	Hexachlorobutadiene	µg/kg	2810	3860	680 - 4280	Acceptable	EPA 8270C
6285	Hexachlorocyclopentadiene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
4840	Hexachloroethane	µg/kg	1250	1830	194 - 1950	Acceptable	EPA 8270C
6315	Indeno(1,2,3-cd)pyrene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6320	Isophorone	µg/kg	7290	9690	840 - 12400	Acceptable	EPA 8270C
6385	2-Methylnaphthalene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6400	2-Methylphenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6412	3 & 4-Methylphenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5005	Naphthalene	µg/kg	1250	1610	371 - 1890	Acceptable	EPA 8270C
6460	2-Nitroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6465	3-Nitroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6470	4-Nitroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5015	Nitrobenzene	µg/kg	2250	3100	492 - 3710	Acceptable	EPA 8270C
6490	2-Nitrophenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6495	3-Nitrophenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6500	4-Nitrophenol	µg/kg	1990	3560	0.00 - 4880	Acceptable	EPA 8270C
6530	N-Nitrosodimethylamine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6535	N-Nitrosodiphenylamine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6545	N-Nitroso-di-n-propylamine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6605	Pentachlorophenol	µg/kg	2970	5810	60.0 - 6540	Acceptable	EPA 8270C
6615	Phenanthrene	µg/kg	1870	2320	840 - 3000	Acceptable	EPA 8270C
6625	Phenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6665	Pyrene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5095	Pyridine	µg/kg	< 660	0.00		Acceptable	EPA 8270C
5155	1,2,4-Trichlorobenzene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6835	2,4,5-Trichlorophenol	µg/kg	3100	6290	390 - 7110	Acceptable	EPA 8270C
6840	2,4,6-Trichlorophenol	µg/kg	2560	4910	422 - 5140	Acceptable	EPA 8270C

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-44 Final Complete Report

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Analytical Management Laboratories
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/30/03
Study Dates: 10/24/03 - 12/08/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil							
5145	2-Amino-1-methylbenzene (o-toluidine)	µg/kg		0.00			
5625	Benzotrichloride	µg/kg		0.00			
5635	Benzyl chloride	µg/kg		0.00			
6350	bis(2-methoxyethyl)phthalate	µg/kg		0.00			
5810	2-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5815	3-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5820	4-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5885	Diamylphthalate	µg/kg		0.00			
5920	2,4-Dibromophenyl-4-nitrophenylether	µg/kg		0.00			
6010	2,4-Dichlorophenyl-3-methyl-4-nitrophenylether	µg/kg		0.00			
6015	2,3-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6020	2,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6025	2,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6030	2,6-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6035	3,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6040	3,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6055	Dicyclohexylphthalate	µg/kg		0.00			
6505	4-Nitrophenylphenylether	µg/kg		0.00			
6195	Dinonylphthalate	µg/kg		0.00			
6280	Hexachlorocyclohexane	µg/kg		0.00			
6305	Hexyl-2-ethylhexylphthalate	µg/kg		0.00			
6335	Maleic anhydride	µg/kg		0.00			
6590	Pentachlorobenzene	µg/kg		0.00			
6595	Pentachlorohexane	µg/kg		0.00			
6717	Tetrachlorobenzenes	µg/kg		0.00			
6705	1,2,3,4-Tetrachlorobenzene	µg/kg		0.00			
6710	1,2,3,5-Tetrachlorobenzene	µg/kg		0.00			
6715	1,2,4,5-Tetrachlorobenzene	µg/kg		0.00			
6730	2,3,4,5-Tetrachlorophenol	µg/kg		0.00			
6735	2,3,4,6-Tetrachlorophenol	µg/kg		0.00			
6740	2,3,5,6-Tetrachlorophenol	µg/kg		0.00			
5150	1,2,3-Trichlorobenzene	µg/kg		0.00			
6800	1,3,5-Trichlorobenzene	µg/kg		0.00			
6845	2,3,4-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6850	2,3,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6855	2,3,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6860	2,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Labcode: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-44 Final Complete Report

Tenkasi S Viswanathan
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Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/30/03
Study Dates: 10/24/03 - 12/08/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil (Cont.)							
6865	2,4,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6870	3,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
Pesticides in Soil							
7025	Aldrin	µg/kg	322	352	150 - 484	Acceptable	EPA 8081A
7110	alpha-BHC	µg/kg	214	267	95.2 - 345	Acceptable	EPA 8081A
7115	beta-BHC	µg/kg	88.3	123	22.0 - 184	Acceptable	EPA 8081A
7105	delta-BHC	µg/kg	136	151	38.1 - 208	Acceptable	EPA 8081A
7120	gamma-BHC(Lindane)	µg/kg	123	155	49.9 - 210	Acceptable	EPA 8081A
7240	alpha-Chlordane	µg/kg	57.1	79.1	25.2 - 108	Acceptable	EPA 8081A
7245	gamma-Chlordane	µg/kg	66.6	91.1	31.2 - 116	Acceptable	EPA 8081A
7355	4,4'-DDD	µg/kg	227	285	115 - 375	Acceptable	EPA 8081A
7360	4,4'-DDE	µg/kg	205	256	122 - 348	Acceptable	EPA 8081A
7365	4,4'-DDT	µg/kg	257	357	90.2 - 502	Acceptable	EPA 8081A
7470	Dieldrin	µg/kg	67.6	77.9	27.9 - 110	Acceptable	EPA 8081A
7540	Endrin	µg/kg	87.3	117	46.8 - 163	Acceptable	EPA 8081A
7530	Endrin aldehyde	µg/kg	61.6	100	16.0 - 132	Acceptable	EPA 8081A
7535	Endrin ketone	µg/kg	234	168	38.2 - 246	Acceptable	EPA 8081A
7510	Endosulfan I	µg/kg	230	311	113 - 387	Acceptable	EPA 8081A
7515	Endosulfan II	µg/kg	183	272	72.8 - 309	Acceptable	EPA 8081A
7520	Endosulfan sulfate	µg/kg	440	392	91.0 - 547	Acceptable	EPA 8081A
7685	Heptachlor	µg/kg	60.2	83.8	23.5 - 120	Acceptable	EPA 8081A
7690	Heptachlor epoxide	µg/kg	166	206	83.9 - 256	Acceptable	EPA 8081A
7810	Methoxychlor	µg/kg	266	287	34.8 - 427	Acceptable	EPA 8081A
Chlordane in Soil							
7250	Chlordane, Technical	µg/kg	210	280	83.3 - 359	Acceptable	EPA 8081A
Toxaphene in Soil							
8250	Toxaphene	µg/kg	318	376	10.3 - 538	Acceptable	EPA 8081A
Volatiles in Soil							
4315	Acetone	µg/kg	30.8	155	0.00 - 306	Acceptable	EPA 8260B
4320	Acetonitrile	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4325	Acrolein	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4375	Benzene	µg/kg	161	162	96.7 - 217	Acceptable	EPA 8260B
4395	Bromodichloromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4400	Bromoform	µg/kg	119	114	62.9 - 163	Acceptable	EPA 8260B

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-44 Final Complete Report

Tenkasi S Viswanathan
QA Director
Analytical Management Laboratories
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/30/03
Study Dates: 10/24/03 - 12/08/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)							
4950	Bromomethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4410	2-Butanone (MEK)	µg/kg	9.90	59.2	0.00 - 97.5	Acceptable	EPA 8260B
4450	Carbon disulfide	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4455	Carbon tetrachloride	µg/kg	68.2	72.0	39.9 - 99.9	Acceptable	EPA 8260B
4475	Chlorobenzene	µg/kg	69.1	69.2	38.1 - 94.1	Acceptable	EPA 8260B
4575	Chlorodibromomethane	µg/kg	167	167	88.5 - 236	Acceptable	EPA 8260B
4485	Chloroethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4500	2-Chloroethylvinylether	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4505	Chloroform	µg/kg	59.3	61.8	41.1 - 80.3	Acceptable	EPA 8260B
4960	Chloromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4570	1,2-Dibromo-3-chloropropane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4585	1,2-Dibromoethane (EDB)	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4595	Dibromomethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4610	1,2-Dichlorobenzene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4615	1,3-Dichlorobenzene	µg/kg	39.1	40.6	12.7 - 58.7	Acceptable	EPA 8260B
4620	1,4-Dichlorobenzene	µg/kg	136	142	42.5 - 202	Acceptable	EPA 8260B
4625	Dichlorodifluoromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4630	1,1-Dichloroethane	µg/kg	123	125	75.4 - 167	Acceptable	EPA 8260B
4635	1,2-Dichloroethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4640	1,1-Dichloroethylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4645	cis-1,2-Dichloroethylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4700	trans-1,2-Dichloroethylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4655	1,2-Dichloropropane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4680	cis-1,3-Dichloropropylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4685	trans-1,3-Dichloropropylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4765	Ethylbenzene	µg/kg	68.8	71.0	40.7 - 97.3	Acceptable	EPA 8260B
4860	2-Hexanone	µg/kg	73.1	76.6	37.9 - 102	Acceptable	EPA 8260B
4975	Methylene chloride	µg/kg	69.2	73.9	36.7 - 108	Acceptable	EPA 8260B
4995	4-Methyl-2-pentanone (MIBK)	µg/kg	91.9	96.9	38.4 - 139	Acceptable	EPA 8260B
5100	Styrene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5105	1,1,1,2-Tetrachloroethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5110	1,1,2,2-Tetrachloroethane	µg/kg	99.7	95.1	43.7 - 139	Acceptable	EPA 8260B
5115	Tetrachloroethylene	µg/kg	131	135	54.8 - 195	Acceptable	EPA 8260B
5140	Toluene	µg/kg	49.9	53.6	32.8 - 70.3	Acceptable	EPA 8260B
5160	1,1,1-Trichloroethane	µg/kg	34.3	35.7	24.4 - 47.4	Acceptable	EPA 8260B
5165	1,1,2-Trichloroethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5170	Trichloroethylene	µg/kg	89.4	102	61.1 - 136	Acceptable	EPA 8260B

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-44 Final Complete Report

Tenkasi S Viswanathan
QA Director
Analytical Management Laboratories
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/30/03
Study Dates: 10/24/03 - 12/08/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)							
5175	Trichlorofluoromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5180	1,2,3-Trichloropropane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5225	Vinyl acetate	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5235	Vinyl chloride	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5260	Total, Xylenes	µg/kg	242	244	114 - 348	Acceptable	EPA 8260B
Herbicides in Soil							
8545	2,4-D	µg/kg	66.2	265	0.00 - 492	Acceptable	EPA 8151A
8555	Dalapon	µg/kg	< 41	0.00		Acceptable	EPA 8151A
8560	2,4-DB	µg/kg	162	371	3.50 - 547	Acceptable	EPA 8151A
8595	Dicamba	µg/kg	111	318	0.00 - 520	Acceptable	EPA 8151A
8620	Dinoseb	µg/kg	98.6	459	0.00 - 682	Acceptable	EPA 8151A
7780	MCPP	µg/kg	< 333	0.00		Acceptable	EPA 8151A
6605	Pentachlorophenol	µg/kg		694	0.00 - 1230		
8655	2,4,5-T	µg/kg	19.4	312	0.00 - 541	Acceptable	EPA 8151A
8650	2,4,5-TP (Silvex)	µg/kg	58.8	201	0.00 - 368	Acceptable	EPA 8151A
PCBs in Soil							
8880	Aroclor 1016	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8885	Aroclor 1221	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8890	Aroclor 1232	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8895	Aroclor 1242	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8900	Aroclor 1248	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8905	Aroclor 1254	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8910	Aroclor 1260	mg/kg	2.72	3.30	1.06 - 4.28	Acceptable	EPA 8082A

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

Study: **SOIL-44**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Laboratories**

Report Type: **Complete**

Report Method: **Method B**

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-44 Final Complete Report

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Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/30/03
Study Dates: 10/24/03 - 12/08/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil							
1000	Aluminum	mg/kg	4720	7260	3880 - 11400	Acceptable	EPA 6020
1005	Antimony	mg/kg	197	223	0.00 - 223	Acceptable	EPA 6020
1010	Arsenic	mg/kg	92.5	101	72.7 - 119	Acceptable	EPA 6020
1015	Barium	mg/kg	136	145	121 - 193	Acceptable	EPA 6020
1020	Beryllium	mg/kg	48.3	54.4	39.8 - 61.2	Acceptable	EPA 6020
1025	Boron	mg/kg	60.7	144	44.4 - 144	Acceptable	EPA 6020
1030	Cadmium	mg/kg	55.1	64.4	42.5 - 66.5	Acceptable	EPA 6020
1035	Calcium	mg/kg	3080	3490	2950 - 4650	Acceptable	EPA 6020
1040	Chromium	mg/kg	60.4	71.9	50.5 - 85.5	Acceptable	EPA 6020
1050	Cobalt	mg/kg	73.3	98.1	58.8 - 98.1	Acceptable	EPA 6020
1055	Copper	mg/kg	75.5	81.7	65.1 - 101	Acceptable	EPA 6020
1070	Iron	mg/kg	8540	10800	3960 - 19400	Acceptable	EPA 6020
1075	Lead	mg/kg	123	126	93.5 - 150	Acceptable	EPA 6020
1085	Magnesium	mg/kg	2130	2600	1960 - 3460	Acceptable	EPA 6020
1090	Manganese	mg/kg	458	465	361 - 571	Acceptable	EPA 6020
1095	Mercury	mg/kg	4.13	5.82	2.74 - 7.26	Acceptable	EPA 6020
1100	Molybdenum	mg/kg	23.7	25.9	15.4 - 31.2	Acceptable	EPA 6020
1105	Nickel	mg/kg	44.5	58.3	41.3 - 66.7	Acceptable	EPA 6020
1125	Potassium	mg/kg	1540	1980	1470 - 2880	Acceptable	EPA 6020
1140	Selenium	mg/kg	103	111	73.7 - 132	Acceptable	EPA 6020
1150	Silver	mg/kg	83.0	114	75.4 - 135	Acceptable	EPA 6020
1155	Sodium	mg/kg	181	206	97.1 - 375	Acceptable	EPA 6020
1160	Strontium	mg/kg	41.4	41.7	34.7 - 69.7	Acceptable	EPA 6020
1165	Thallium	mg/kg	193	199	135 - 229	Acceptable	EPA 6020
1175	Tin	mg/kg	96.9	164	68.4 - 164	Acceptable	EPA 6020
1180	Titanium	mg/kg	174	338	0.00 - 661	Acceptable	EPA 6020
1185	Vanadium	mg/kg	98.5	122	84.2 - 148	Acceptable	EPA 6020
1190	Zinc	mg/kg	223	247	181 - 303	Acceptable	EPA 6020

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES® SOIL-45 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 **EPA ID: KS00902**

Report Issued: 03/29/04
Study Dates: 01/26/04 - 03/11/04

SOIL Study Definitions:

The reported value is the value that the laboratory reported to ERA.

The ERA assigned value for the Organic Proficiency Testing Standards is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation as applicable. The ERA assigned value for the Inorganic Proficiency Testing Standards is equal to the maximum amount of the parameter available in the standard by applicable EPA methodologies. All NELAC parameters not added to a standard are given an assigned Value of "0", per the guidance issued by the NELAC Board of Directors, on December 14, 2000. Non-NELAC parameters not added to a standard may be given an assigned value of less than a minimum verified concentration as determined in the background soil for applicable EPA methodologies.

The acceptance limits are established per the NELAC PT program criteria or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For a further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable = Reported Value falls within the Acceptance Limits.

Not Acceptable = Reported Value falls outside of the Acceptance Limits.

No Evaluation = Reported Value cannot be evaluated.

The method description is the method the laboratory reported to ERA.

SOIL Study Discussion:

ERA's Proficiency Testing (PT) Study, SOIL-45, has been reviewed by ERA Senior Management and certified compliant with the requirements of the National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program Standards, Chapter 2, July 2003.

Per the requirements of the NELAC Proficiency Testing Program, a full review of all accuracy verification, homogeneity and stability data was completed. All analytical verification data for all analytes in the SOIL-45 standards met the acceptance criteria contained in the NELAC Proficiency Testing Program Standards, Chapter 2, July 2003.

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's review of the data no anomalies were noted.

The Proficiency Testing Study, SOIL-45, report shall not be reproduced except in its entirety and not without the permission of the participating laboratory. The report must not be used by the participating laboratory to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding ERA's Proficiency Testing Study, SOIL-45, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

Study: **SOIL-45**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method A**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-45 Final Complete Report

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913-829-0101

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 03/29/04
Study Dates: 01/26/04 - 03/11/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Nitroaromatics & Nitramines in Soil							
9306	4-Amino-2,6-dinitrotoluene	µg/kg	< 100	0.00		Acceptable	EPA 8330
9303	2-Amino-4,6-dinitrotoluene	µg/kg	2700	3070	1140 - 3780	Acceptable	EPA 8330
6160	1,3-Dinitrobenzene	µg/kg	< 100	0.00		Acceptable	EPA 8330
6185	2,4-Dinitrotoluene	µg/kg	7900	7780	3790 - 10600	Acceptable	EPA 8330
6190	2,6-Dinitrotoluene	µg/kg	8370	8390	2670 - 12300	Acceptable	EPA 8330
9522	HMX	µg/kg	5520	5600	1360 - 7180	Acceptable	EPA 8330
5015	Nitrobenzene	µg/kg	21000	21700	0.00 - 34700	Acceptable	EPA 8330
9507	2-Nitrotoluene	µg/kg	< 100	0.00		Acceptable	EPA 8330
9510	3-Nitrotoluene	µg/kg	< 100	0.00		Acceptable	EPA 8330
9513	4-Nitrotoluene	µg/kg	< 100	0.00		Acceptable	EPA 8330
9432	RDX	µg/kg	3530	3870	1930 - 4670	Acceptable	EPA 8330
6415	Tetryl	µg/kg	< 100	0.00		Acceptable	EPA 8330
6885	1,3,5-Trinitrobenzene	µg/kg	16700	16700	0.00 - 26900	Acceptable	EPA 8330
9651	2,4,6-Trinitrotoluene	µg/kg	12100	13300	7300 - 15100	Acceptable	EPA 8330

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

SOIL-46 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 **EPA ID: KS00902**

Report Issued: 07/01/04
Study Dates: 04/26/04 - 06/10/04

SOIL Study Definitions:

The reported value is the value that the laboratory reported to ERA.

The ERA assigned value for the Organic Proficiency Testing Standards is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation as applicable. The ERA assigned value for the Inorganic Proficiency Testing Standards is equal to the maximum amount of the parameter available in the standard by applicable EPA methodologies. All NELAC parameters not added to a standard are given an assigned Value of "0", per the guidance issued by the NELAC Board of Directors, on December 14, 2000. Non-NELAC parameters not added to a standard may be given an assigned value of less than a minimum verified concentration as determined in the background soil for applicable EPA methodologies.

The acceptance limits are established per the NELAC PT program criteria or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For a further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable = Reported Value falls within the Acceptance Limits.

Not Acceptable = Reported Value falls outside of the Acceptance Limits.

No Evaluation = Reported Value cannot be evaluated.

The method description is the method the laboratory reported to ERA.

SOIL Study Discussion:

ERA's Proficiency Testing (PT) Study, SOIL-46, has been reviewed by ERA Senior Management and certified compliant with the requirements of the National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program Standards, Chapter 2, July 2003.

Per the requirements of the NELAC Proficiency Testing Program, a full review of all accuracy verification, homogeneity and stability data was completed. All analytical verification data for all analytes in the SOIL-46 standards met the acceptance criteria contained in the NELAC Proficiency Testing Program Standards, Chapter 2, July 2003.

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's review of the data one anomaly was noted. This anomaly is addressed on the following page.

The Proficiency Testing Study, SOIL-46, report shall not be reproduced except in its entirety and not without the permission of the participating laboratory. The report must not be used by the participating laboratory to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding ERA's Proficiency Testing Study, SOIL-46, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

ERA Laboratory Code: A5230-01 **EPA ID: KS00902**

Report Issued: 07/01/04
Study Dates: 04/26/04 - 06/10/04

Study Discussion: PCBs in Soil

During the review of the SOIL-46 PCBs in Soil data set, ERA observed a failure rate of 15.1% for Aroclor 1242. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification and stability data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'.

Upon further examination of the PCBs in Soil data set, ERA found that three laboratories misidentified Aroclor 1242 as Aroclor 1232, five laboratories misidentified Aroclor 1242 as Aroclor 1248 and one laboratory misidentified Aroclor 1242 as Aroclor 1254. The failure rate for Aroclor 1242 without the apparent aroclor misidentifications is 6.25%. This failure rate is more consistent with ERA's historical failure rate of 6.00%.

If you are one of the laboratories that misidentified Aroclor 1242 and would like technical assistance please call ERA's Organic Chemistry Department at 1-800-372-0122.

NVLAP® Lab code: 200386-0

Study: **SOIL-46**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method A**

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-46 Final Complete Report

Tenkasi Viswanathan
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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 07/01/04
Study Dates: 04/26/04 - 06/10/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil							
1000	Aluminum	mg/kg	3860	7060	2330 - 12200	Acceptable	EPA 6020
1005	Antimony	mg/kg	167	200	0.00 - 200	Acceptable	EPA 6020
1010	Arsenic	mg/kg	92.2	99.0	63.2 - 119	Acceptable	EPA 6020
1015	Barium	mg/kg	236	274	198 - 326	Acceptable	EPA 6020
1020	Beryllium	mg/kg	60.5	65.8	43.7 - 79.1	Acceptable	EPA 6020
1025	Boron	mg/kg	98.8	123	63.2 - 145	Acceptable	EPA 6020
1030	Cadmium	mg/kg	69.9	81.1	51.7 - 92.3	Acceptable	EPA 6020
1035	Calcium	mg/kg	3470	3860	2700 - 4660	Acceptable	EPA 6020
1040	Chromium	mg/kg	82.2	93.9	57.1 - 111	Acceptable	EPA 6020
1050	Cobalt	mg/kg	114	116	82.3 - 142	Acceptable	EPA 6020
1055	Copper	mg/kg	67.5	68.6	50.9 - 83.3	Acceptable	EPA 6020
1070	Iron	mg/kg	11700	16600	1780 - 26000	Acceptable	EPA 6020
1075	Lead	mg/kg	114	126	88.4 - 150	Acceptable	EPA 6020
1085	Magnesium	mg/kg	1770	2290	1550 - 2990	Acceptable	EPA 6020
1090	Manganese	mg/kg	198	218	159 - 267	Acceptable	EPA 6020
1095	Mercury	mg/kg	2.09	2.18	1.36 - 3.28	Acceptable	EPA 6020
1100	Molybdenum	mg/kg	51.3	55.8	31.3 - 65.7	Acceptable	EPA 6020
1105	Nickel	mg/kg	85.7	97.6	63.4 - 111	Acceptable	EPA 6020
1125	Potassium	mg/kg	1840	2310	1510 - 3410	Acceptable	EPA 6020
1140	Selenium	mg/kg	133	140	87.8 - 168	Acceptable	EPA 6020
1150	Silver	mg/kg	128	127	65.1 - 163	Acceptable	EPA 6020
1155	Sodium	mg/kg	202	235	111 - 355	Acceptable	EPA 6020
1160	Strontium	mg/kg	37.8	48.4	30.6 - 64.0	Acceptable	EPA 6020
1165	Thallium	mg/kg	133	157	93.9 - 188	Acceptable	EPA 6020
1175	Tin	mg/kg	111	125	72.0 - 150	Acceptable	EPA 6020
1180	Titanium	mg/kg	158	280	0.00 - 596	Acceptable	EPA 6020
1185	Vanadium	mg/kg	103	121	67.9 - 138	Acceptable	EPA 6020
1190	Zinc	mg/kg	291	307	201 - 379	Acceptable	EPA 6020
Cyanide in Soil							
1635	Cyanide, total	mg/kg	123	184	0.00 - 272	Acceptable	EPA 9010B
1640	Reactive Cyanide	mg/kg	< 25	< 25.0	< 25.0	Acceptable	EPA 9010B
Hexavalent Chromium in Soil							
1045	Chromium VI	mg/kg	111	272	13.5 - 272	Acceptable	EPA 7196A

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

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SOIL-46 Final Complete Report

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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 07/01/04
Study Dates: 04/26/04 - 06/10/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil							
5500	Acenaphthene	µg/kg	4810	5820	1720 - 7120	Acceptable	EPA 8310 UV
5505	Acenaphthylene	µg/kg	1300	1410	360 - 1680	Acceptable	EPA 8310 UV
5545	Aniline	µg/kg		0.00			
5555	Anthracene	µg/kg	< 125	0.00		Acceptable	EPA 8310 UV
5595	Benzidine	µg/kg		0.00			
5610	Benzoic acid	µg/kg		0.00			
5575	Benzo(a)anthracene	µg/kg	< 125	0.00		Acceptable	EPA 8310 UV
5585	Benzo(b)fluoranthene	µg/kg	2820	2820	1030 - 3650	Acceptable	EPA 8310 UV
5600	Benzo(k)fluoranthene	µg/kg	< 125	0.00		Acceptable	EPA 8310 UV
5590	Benzo(g,h,i)peryiene	µg/kg	1530	1930	598 - 2480	Acceptable	EPA 8310 UV
5580	Benzo(a)pyrene	µg/kg	< 125	0.00		Acceptable	EPA 8310 UV
5630	Benzyl alcohol	µg/kg		0.00			
5660	4-Bromophenyl-phenylether	µg/kg		0.00			
5670	Butylbenzylphthalate	µg/kg		7980	1640 - 11300		
5680	Carbazole	µg/kg		0.00			
5745	4-Chloroaniline	µg/kg		0.00			
5760	bis(2-Chloroethoxy)methane	µg/kg		4080	675 - 5090		
5765	bis(2-Chloroethyl)ether	µg/kg		0.00			
5780	bis(2-Chloroisopropyl)ether	µg/kg		7380	610 - 9310		
5700	4-Chloro-3-methylphenol	µg/kg		8970	970 - 10300		
5790	1-Chloronaphthalene	µg/kg		0.00			
5795	2-Chloronaphthalene	µg/kg		8320	1810 - 10200		
5800	2-Chlorophenol	µg/kg		8800	490 - 9550		
5825	4-Chlorophenyl-phenylether	µg/kg		0.00			
5855	Chrysene	µg/kg	4150	5300	2020 - 6780	Acceptable	EPA 8310 UV
5895	Dibenz(a,h)anthracene	µg/kg	3620	5040	1410 - 6770	Acceptable	EPA 8310 UV
5905	Dibenzofuran	µg/kg		5590	1910 - 6710		
5925	Di-n-butylphthalate	µg/kg		4910	1510 - 6490		
4610	1,2-Dichlorobenzene	µg/kg		0.00			
4615	1,3-Dichlorobenzene	µg/kg		0.00			
4620	1,4-Dichlorobenzene	µg/kg		8860	0.00 - 10000		
5945	3,3'-Dichlorobenzidine	µg/kg		0.00			
6000	2,4-Dichlorophenol	µg/kg		3460	184 - 3980		
6005	2,6-Dichlorophenol	µg/kg		0.00			
6070	Diethylphthalate	µg/kg		7200	1490 - 9470		
6130	2,4-Dimethylphenol	µg/kg		0.00			
6135	Dimethylphthalate	µg/kg		5460	1370 - 6860		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-46 Final Complete Report

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)							
6360	4,6-Dinitro-2-methylphenol	µg/kg		8480	0.00 - 9170		
6175	2,4-Dinitrophenol	µg/kg		0.00			
6185	2,4-Dinitrotoluene	µg/kg		9680	1310 - 12700		
6190	2,6-Dinitrotoluene	µg/kg		2510	799 - 3120		
6200	Di-n-octylphthalate	µg/kg		0.00			
6255	bis(2-Ethylhexyl)phthalate	µg/kg		7000	1950 - 9450		
6265	Fluoranthene	µg/kg	6840	8980	3310 - 11300	Acceptable	EPA 8310 UV
6270	Fluorene	µg/kg	< 125	0.00		Acceptable	EPA 8310 UV
6275	Hexachlorobenzene	µg/kg		2410	908 - 3030		
4835	Hexachlorobutadiene	µg/kg		0.00			
6285	Hexachlorocyclopentadiene	µg/kg		0.00			
4840	Hexachloroethane	µg/kg		0.00			
6315	Indeno(1,2,3-cd)pyrene	µg/kg	< 125	0.00		Acceptable	EPA 8310 UV
6320	Isophorone	µg/kg		5700	640 - 7300		
6385	2-Methylnaphthalene	µg/kg		0.00			
6400	2-Methylphenol	µg/kg		9000	230 - 9000		
6412	3 & 4-Methylphenol	µg/kg		0.00			
5005	Naphthalene	µg/kg	4370	6690	1030 - 7990	Acceptable	EPA 8310 UV
6460	2-Nitroaniline	µg/kg		0.00			
6465	3-Nitroaniline	µg/kg		0.00			
6470	4-Nitroaniline	µg/kg		0.00			
5015	Nitrobenzene	µg/kg		0.00			
6490	2-Nitrophenol	µg/kg		5200	233 - 6110		
6500	4-Nitrophenol	µg/kg		7280	0.00 - 9370		
6530	N-Nitrosodimethylamine	µg/kg		4730	0.00 - 6190		
6535	N-Nitrosodiphenylamine	µg/kg		0.00			
6545	N-Nitroso-di-n-propylamine	µg/kg		1500	102 - 2060		
6605	Pentachlorophenol	µg/kg		0.00			
6615	Phenanthrene	µg/kg	3450	3650	1600 - 4520	Acceptable	EPA 8310 UV
6625	Phenol	µg/kg		5750	457 - 6400		
6665	Pyrene	µg/kg	2570	2850	1030 - 3750	Acceptable	EPA 8310 UV
5095	Pyridine	µg/kg		0.00			
5155	1,2,4-Trichlorobenzene	µg/kg		0.00			
6835	2,4,5-Trichlorophenol	µg/kg		8390	600 - 9720		
6840	2,4,6-Trichlorophenol	µg/kg		9400	680 - 10800		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 07/01/04
Study Dates: 04/26/04 - 06/10/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil							
5145	2-Amino-1-methylbenzene (o-toluidine)	µg/kg		0.00			
5625	Benzotrichloride	µg/kg		0.00			
5635	Benzyl chloride	µg/kg		0.00			
6350	bis(2-methoxyethyl)phthalate	µg/kg		0.00			
5810	2-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5815	3-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5820	4-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5885	Diamylphthalate	µg/kg		0.00			
5920	2,4-Dibromophenyl-4-nitrophenylether	µg/kg		0.00			
6010	2,4-Dichlorophenyl-3-methyl-4-nitrophenylether	µg/kg		0.00			
6015	2,3-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6020	2,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6025	2,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6030	2,6-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6035	3,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6040	3,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6055	Dicyclohexylphthalate	µg/kg		0.00			
6505	4-Nitrophenylphenylether	µg/kg		0.00			
6195	Dinonylphthalate	µg/kg		0.00			
6280	Hexachlorocyclohexane	µg/kg		0.00			
6305	Hexyl-2-ethylhexylphthalate	µg/kg		0.00			
6335	Maleic anhydride	µg/kg		0.00			
6590	Pentachlorobenzene	µg/kg		0.00			
6595	Pentachlorohexane	µg/kg		0.00			
6717	Tetrachlorobenzenes	µg/kg		0.00			
6705	1,2,3,4-Tetrachlorobenzene	µg/kg		0.00			
6710	1,2,3,5-Tetrachlorobenzene	µg/kg		0.00			
6715	1,2,4,5-Tetrachlorobenzene	µg/kg		0.00			
6730	2,3,4,5-Tetrachlorophenol	µg/kg		0.00			
6735	2,3,4,6-Tetrachlorophenol	µg/kg		0.00			
6740	2,3,5,6-Tetrachlorophenol	µg/kg		0.00			
5150	1,2,3-Trichlorobenzene	µg/kg		0.00			
6800	1,3,5-Trichlorobenzene	µg/kg		0.00			
6845	2,3,4-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6850	2,3,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6855	2,3,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6860	2,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-46 Final Complete Report

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil (Cont.)							
6865	2,4,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6870	3,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
Pesticides in Soil							
7025	Aldrin	µg/kg	199	201	85.8 - 298	Acceptable	EPA 8081A
7110	alpha-BHC	µg/kg	111	137	44.8 - 197	Acceptable	EPA 8081A
7115	beta-BHC	µg/kg	105	155	26.0 - 224	Acceptable	EPA 8081A
7105	delta-BHC	µg/kg	273	329	87.1 - 461	Acceptable	EPA 8081A
7120	gamma-BHC(Lindane)	µg/kg	184	211	85.4 - 279	Acceptable	EPA 8081A
7240	alpha-Chlordane	µg/kg	161	190	67.9 - 258	Acceptable	EPA 8081A
7245	gamma-Chlordane	µg/kg	121	152	54.6 - 203	Acceptable	EPA 8081A
7355	4,4'-DDD	µg/kg	112	142	58.5 - 206	Acceptable	EPA 8081A
7360	4,4'-DDE	µg/kg	108	151	50.4 - 226	Acceptable	EPA 8081A
7365	4,4'-DDT	µg/kg	99.0	123	37.2 - 185	Acceptable	EPA 8081A
7470	Dieldrin	µg/kg	77.9	96.7	43.2 - 133	Acceptable	EPA 8081A
7540	Endrin	µg/kg	125	133	61.6 - 198	Acceptable	EPA 8081A
7530	Endrin aldehyde	µg/kg	50.4	207	0.00 - 243	Acceptable	EPA 8081A
7535	Endrin ketone	µg/kg	189	232	78.1 - 320	Acceptable	EPA 8081A
7510	Endosulfan I	µg/kg	236	302	94.9 - 381	Acceptable	EPA 8081A
7515	Endosulfan II	µg/kg	157	231	60.2 - 292	Acceptable	EPA 8081A
7520	Endosulfan sulfate	µg/kg	180	205	49.5 - 311	Acceptable	EPA 8081A
7685	Heptachlor	µg/kg	174	196	72.0 - 270	Acceptable	EPA 8081A
7690	Heptachlor epoxide	µg/kg	129	149	53.0 - 209	Acceptable	EPA 8081A
7810	Methoxychlor	µg/kg	356	394	37.0 - 643	Acceptable	EPA 8081A
Chlordane in Soil							
7250	Chlordane, Technical	µg/kg	260	420	51.9 - 548	Acceptable	EPA 8081A
Toxaphene in Soil							
8250	Toxaphene	µg/kg	225	375	0.00 - 492	Acceptable	EPA 8081A
Volatiles in Soil							
4315	Acetone	µg/kg	127	170	0.00 - 295	Acceptable	EPA 8260B
4320	Acetonitrile	µg/kg		0.00			
4325	Acrolein	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4375	Benzene	µg/kg	96.6	95.8	58.8 - 134	Acceptable	EPA 8260B
4395	Bromodichloromethane	µg/kg	33.3	30.7	18.9 - 45.1	Acceptable	EPA 8260B
4400	Bromoform	µg/kg	65.5	60.8	35.0 - 90.6	Acceptable	EPA 8260B

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)							
4950	Bromomethane	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4410	2-Butanone (MEK)	µg/kg	51.3	140	8.50 - 246	Acceptable	EPA 8260B
4450	Carbon disulfide	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4455	Carbon tetrachloride	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4475	Chlorobenzene	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4575	Chlorodibromomethane	µg/kg	80.5	72.8	47.2 - 100	Acceptable	EPA 8260B
4485	Chloroethane	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4500	2-Chloroethylvinylether	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4505	Chloroform	µg/kg	80.7	78.0	49.5 - 113	Acceptable	EPA 8260B
4960	Chloromethane	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4570	1,2-Dibromo-3-chloropropane (DBCP)	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4585	1,2-Dibromoethane (EDB)	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4595	Dibromomethane	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4610	1,2-Dichlorobenzene	µg/kg	75.1	67.8	45.4 - 92.6	Acceptable	EPA 8260B
4615	1,3-Dichlorobenzene	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4620	1,4-Dichlorobenzene	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4625	Dichlorodifluoromethane	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4630	1,1-Dichloroethane	µg/kg	135	141	84.5 - 214	Acceptable	EPA 8260B
4635	1,2-Dichloroethane	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4640	1,1-Dichloroethylene	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4645	cis-1,2-Dichloroethylene	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4700	trans-1,2-Dichloroethylene	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4655	1,2-Dichloropropane	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4680	cis-1,3-Dichloropropylene	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4685	trans-1,3-Dichloropropylene	µg/kg	< 2	0.00		Acceptable	EPA 8260B
4765	Ethylbenzene	µg/kg	118	114	75.9 - 158	Acceptable	EPA 8260B
4860	2-Hexanone	µg/kg	152	136	56.7 - 201	Acceptable	EPA 8260B
4975	Methylene chloride	µg/kg	80.7	83.2	39.5 - 124	Acceptable	EPA 8260B
4995	4-Methyl-2-pentanone (MIBK)	µg/kg	160	140	58.6 - 219	Acceptable	EPA 8260B
5100	Styrene	µg/kg	< 2	0.00		Acceptable	EPA 8260B
5105	1,1,1,2-Tetrachloroethane	µg/kg	63.4	58.8	38.9 - 79.3	Acceptable	EPA 8260B
5110	1,1,2,2-Tetrachloroethane	µg/kg	88.0	69.3	42.9 - 95.1	Acceptable	EPA 8260B
5115	Tetrachloroethylene	µg/kg	106	106	55.5 - 155	Acceptable	EPA 8260B
5140	Toluene	µg/kg	171	178	112 - 240	Acceptable	EPA 8260B
5160	1,1,1-Trichloroethane	µg/kg	78.0	77.8	48.8 - 113	Acceptable	EPA 8260B
5165	1,1,2-Trichloroethane	µg/kg	88.3	81.0	54.5 - 111	Acceptable	EPA 8260B
5170	Trichloroethylene	µg/kg	117	118	70.1 - 174	Acceptable	EPA 8260B

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

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SOIL-46 Final Complete Report

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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 07/01/04
Study Dates: 04/26/04 - 06/10/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)							
5175	Trichlorofluoromethane	µg/kg	< 2	0.00		Acceptable	EPA 8260B
5180	1,2,3-Trichloropropane	µg/kg	< 2	0.00		Acceptable	EPA 8260B
5225	Vinyl acetate	µg/kg	< 2	0.00		Acceptable	EPA 8260B
5235	Vinyl chloride	µg/kg	< 2	0.00		Acceptable	EPA 8260B
5260	Total, Xylenes	µg/kg	207	210	135 - 294	Acceptable	EPA 8260B
Herbicides in Soil							
8545	2,4-D	µg/kg	6.40	338	0.00 - 495	Acceptable	EPA 8151A
8555	Dalapon	µg/kg	< 1.67	0.00		Acceptable	EPA 8151A
8560	2,4-DB	µg/kg	94.8	283	0.00 - 497	Acceptable	EPA 8151A
8595	Dicamba	µg/kg	6.66	292	0.00 - 423	Acceptable	EPA 8151A
8620	Dinoseb	µg/kg	261	479	0.00 - 617	Acceptable	EPA 8151A
7780	MCPP	µg/kg	< 167	0.00		Acceptable	EPA 8151A
6605	Pentachlorophenol	µg/kg	369	856	0.00 - 961	Acceptable	EPA 8151A
8655	2,4,5-T	µg/kg	20.4	196	0.00 - 334	Acceptable	EPA 8151A
8650	2,4,5-TP (Silvex)	µg/kg	31.5	315	0.00 - 468	Acceptable	EPA 8151A
PCBs in Soil							
8880	Aroclor 1016	mg/kg	< 0.0067	0.00		Acceptable	EPA 8082A
8885	Aroclor 1221	mg/kg	< 0.0067	0.00		Acceptable	EPA 8082A
8890	Aroclor 1232	mg/kg	< 0.0067	0.00		Acceptable	EPA 8082A
8895	Aroclor 1242	mg/kg	2.71	7.97	0.750 - 11.4	Acceptable	EPA 8082A
8900	Aroclor 1248	mg/kg	< 0.0067	0.00		Acceptable	EPA 8082A
8905	Aroclor 1254	mg/kg	< 0.0067	0.00		Acceptable	EPA 8082A
8910	Aroclor 1260	mg/kg	< 0.0067	0.00		Acceptable	EPA 8082A

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP®

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-46 Final Complete Report

Tenkasi Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 07/01/04
Study Dates: 04/26/04 - 06/10/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
PAHs in Soil							
5500	Acenaphthene	µg/kg	109	314	0.00 - 424	Acceptable	EPA 8270 SIM
5505	Acenaphthylene	µg/kg	54.3	214	0.00 - 238	Acceptable	EPA 8270 SIM
5555	Anthracene	µg/kg	14.2	90.8	0.00 - 90.8	Acceptable	EPA 8270 SIM
5575	Benzo(a)anthracene	µg/kg	35.7	79.7	0.00 - 115	Acceptable	EPA 8270 SIM
5585	Benzo(b)fluoranthene	µg/kg	44.5	78.4	0.00 - 133	Acceptable	EPA 8270 SIM
5600	Benzo(k)fluoranthene	µg/kg	41.2	74.5	0.00 - 116	Acceptable	EPA 8270 SIM
5590	Benzo(g,h,i)perylene	µg/kg	28.7	50.5	0.00 - 88.9	Acceptable	EPA 8270 SIM
5580	Benzo(a)pyrene	µg/kg	29.3	84.3	0.00 - 123	Acceptable	EPA 8270 SIM
5855	Chrysene	µg/kg	44.3	94.6	0.00 - 152	Acceptable	EPA 8270 SIM
5895	Dibenz(a,h)anthracene	µg/kg	19.7	44.1	0.00 - 66.0	Acceptable	EPA 8270 SIM
6265	Fluoranthene	µg/kg	25.9	53.3	0.00 - 106	Acceptable	EPA 8270 SIM
6270	Fluorene	µg/kg	75.3	221	0.00 - 303	Acceptable	EPA 8270 SIM
6315	Indeno(1,2,3-cd)pyrene	µg/kg	35.8	83.3	0.00 - 133	Acceptable	EPA 8270 SIM
5005	Naphthalene	µg/kg	183	419	0.00 - 534	Acceptable	EPA 8270 SIM
6615	Phenanthrene	µg/kg	35.7	89.1	0.00 - 139	Acceptable	EPA 8270 SIM
6665	Pyrene	µg/kg	31.0	60.1	0.00 - 98.2	Acceptable	EPA 8270 SIM
Nitroaromatics & Nitramines in Soil							
9306	4-Amino-2,6-dinitrotoluene	µg/kg	< 100	0.00		Acceptable	EPA 8330
9303	2-Amino-4,6-dinitrotoluene	µg/kg	2510	2800	1090 - 3410	Acceptable	EPA 8330
6160	1,3-Dinitrobenzene	µg/kg	4310	4080	2120 - 5160	Acceptable	EPA 8330
6185	2,4-Dinitrotoluene	µg/kg	10200	9180	4640 - 12600	Acceptable	EPA 8330
6190	2,6-Dinitrotoluene	µg/kg	< 100	0.00		Acceptable	EPA 8330
9522	HMX	µg/kg	< 100	0.00		Acceptable	EPA 8330
5015	Nitrobenzene	µg/kg	6800	6280	3040 - 8400	Acceptable	EPA 8330
9507	2-Nitrotoluene	µg/kg	9740	9510	4610 - 12400	Acceptable	EPA 8330
9510	3-Nitrotoluene	µg/kg	< 100	0.00		Acceptable	EPA 8330
9513	4-Nitrotoluene	µg/kg	9240	8660	3800 - 11700	Acceptable	EPA 8330
9432	RDX	µg/kg	13600	15300	9140 - 16500	Acceptable	EPA 8330
6415	Tetryl	µg/kg	< 100	0.00		Acceptable	EPA 8330
6885	1,3,5-Trinitrobenzene	µg/kg	< 100	0.00		Acceptable	EPA 8330
9651	2,4,6-Trinitrotoluene	µg/kg	5890	6400	3140 - 7180	Acceptable	EPA 8330
Ignitability							
1780	Ignitability (Flashpoint)	deg. F	112	122	108 - 138	Acceptable	EPA 1010
Corrosivity (pH)							
1625	Corrosivity (pH)	S.U.	10.20	9.52	8.89 - 9.79	Not Acceptable	EPA 9045C

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† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

Study: **SOIL-46**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method B**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-46 Final Complete Report

Tenkasi Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 07/01/04
Study Dates: 04/26/04 - 06/10/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil							
1000	Aluminum	mg/kg	4200	7060	2330 - 12200	Acceptable	EPA 6010B
1005	Antimony	mg/kg	178	200	0.00 - 200	Acceptable	EPA 6010B
1010	Arsenic	mg/kg	104	99.0	63.2 - 119	Acceptable	EPA 6010B
1015	Barium	mg/kg	267	274	198 - 326	Acceptable	EPA 6010B
1020	Beryllium	mg/kg	62.0	65.8	43.7 - 79.1	Acceptable	EPA 6010B
1025	Boron	mg/kg		123	63.2 - 145		
1030	Cadmium	mg/kg	78.0	81.1	51.7 - 92.3	Acceptable	EPA 6010B
1035	Calcium	mg/kg	3800	3860	2700 - 4660	Acceptable	EPA 6010B
1040	Chromium	mg/kg	87.1	93.9	57.1 - 111	Acceptable	EPA 6010B
1050	Cobalt	mg/kg	120	116	82.3 - 142	Acceptable	EPA 6010B
1055	Copper	mg/kg	70.4	68.6	50.9 - 83.3	Acceptable	EPA 6010B
1070	Iron	mg/kg	12300	16600	1780 - 26000	Acceptable	EPA 6010B
1075	Lead	mg/kg	130	126	88.4 - 150	Acceptable	EPA 6010B
1085	Magnesium	mg/kg	1920	2290	1550 - 2990	Acceptable	EPA 6010B
1090	Manganese	mg/kg	213	218	159 - 267	Acceptable	EPA 6010B
1095	Mercury	mg/kg	2.26	2.18	1.36 - 3.28	Acceptable	EPA 7471A
1100	Molybdenum	mg/kg	56.3	55.8	31.3 - 65.7	Acceptable	EPA 6010B
1105	Nickel	mg/kg	92.1	97.6	63.4 - 111	Acceptable	EPA 6010B
1125	Potassium	mg/kg	1950	2310	1510 - 3410	Acceptable	EPA 6010B
1140	Selenium	mg/kg	147	140	87.8 - 168	Acceptable	EPA 6010B
1150	Silver	mg/kg	128	127	65.1 - 163	Acceptable	EPA 6010B
1155	Sodium	mg/kg	213	235	111 - 355	Acceptable	EPA 6010B
1160	Strontium	mg/kg		48.4	30.6 - 64.0		
1165	Thallium	mg/kg	145	157	93.9 - 188	Acceptable	EPA 6010B
1175	Tin	mg/kg		125	72.0 - 150		
1180	Titanium	mg/kg		280	0.00 - 596		
1185	Vanadium	mg/kg	107	121	67.9 - 138	Acceptable	EPA 6010B
1190	Zinc	mg/kg	303	307	201 - 379	Acceptable	EPA 6010B

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
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NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-46 Final Complete Report

Tenkasi Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 07/01/04
Study Dates: 04/26/04 - 06/10/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil							
5500	Acenaphthene	µg/kg	4140	5820	1720 - 7120	Acceptable	EPA 8270C
5505	Acenaphthylene	µg/kg	992	1410	360 - 1680	Acceptable	EPA 8270C
5545	Aniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5555	Anthracene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5595	Benzidine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5610	Benzoic acid	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5575	Benzo(a)anthracene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5585	Benzo(b)fluoranthene	µg/kg	2920	2820	1030 - 3650	Acceptable	EPA 8270C
5600	Benzo(k)fluoranthene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5590	Benzo(g,h,i)perylene	µg/kg	1390	1930	598 - 2480	Acceptable	EPA 8270C
5580	Benzo(a)pyrene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5630	Benzyl alcohol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5660	4-Bromophenyl-phenylether	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5670	Butylbenzylphthalate	µg/kg	6150	7980	1640 - 11300	Acceptable	EPA 8270C
5680	Carbazole	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5745	4-Chloroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5760	bis(2-Chloroethoxy)methane	µg/kg	< 125	4080	675 - 5090	Not Acceptable	EPA 8270C
5765	bis(2-Chloroethyl)ether	µg/kg	2990	0.00		Not Acceptable	EPA 8270C
5780	bis(2-Chloroisopropyl)ether	µg/kg	4600	7380	610 - 9310	Acceptable	EPA 8270C
5700	4-Chloro-3-methylphenol	µg/kg	4720	8970	970 - 10300	Acceptable	EPA 8270C
5790	1-Chloronaphthalene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5795	2-Chloronaphthalene	µg/kg	5880	8320	1810 - 10200	Acceptable	EPA 8270C
5800	2-Chlorophenol	µg/kg	4510	8800	490 - 9550	Acceptable	EPA 8270C
5825	4-Chlorophenyl-phenylether	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5855	Chrysene	µg/kg	3630	5300	2020 - 6780	Acceptable	EPA 8270C
5895	Dibenz(a,h)anthracene	µg/kg	3560	5040	1410 - 6770	Acceptable	EPA 8270C
5905	Dibenzofuran	µg/kg	4260	5590	1910 - 6710	Acceptable	EPA 8270C
5925	Di-n-butylphthalate	µg/kg	3870	4910	1510 - 6490	Acceptable	EPA 8270C
4610	1,2-Dichlorobenzene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
4615	1,3-Dichlorobenzene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
4620	1,4-Dichlorobenzene	µg/kg	4920	8860	0.00 - 10000	Acceptable	EPA 8270C
5945	3,3'-Dichlorobenzidine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6000	2,4-Dichlorophenol	µg/kg	1880	3460	184 - 3980	Acceptable	EPA 8270C
6005	2,6-Dichlorophenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6070	Diethylphthalate	µg/kg	5580	7200	1490 - 9470	Acceptable	EPA 8270C
6130	2,4-Dimethylphenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6135	Dimethylphthalate	µg/kg	4170	5460	1370 - 6860	Acceptable	EPA 8270C

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-46 Final Complete Report

Tenkasi Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 07/01/04
Study Dates: 04/26/04 - 06/10/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)							
6360	4,6-Dinitro-2-methylphenol	µg/kg	4730	8480	0.00 - 9170	Acceptable	EPA 8270C
6175	2,4-Dinitrophenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6185	2,4-Dinitrotoluene	µg/kg	7750	9680	1310 - 12700	Acceptable	EPA 8270C
6190	2,6-Dinitrotoluene	µg/kg	1720	2510	799 - 3120	Acceptable	EPA 8270C
6200	Di-n-octylphthalate	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6255	bis(2-Ethylhexyl)phthalate	µg/kg	5420	7000	1950 - 9450	Acceptable	EPA 8270C
6265	Fluoranthene	µg/kg	6890	8980	3310 - 11300	Acceptable	EPA 8270C
6270	Fluorene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6275	Hexachlorobenzene	µg/kg	2060	2410	908 - 3030	Acceptable	EPA 8270C
4835	Hexachlorobutadiene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6285	Hexachlorocyclopentadiene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
4840	Hexachloroethane	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6315	Indeno(1,2,3-cd)pyrene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6320	Isophorone	µg/kg	4210	5700	640 - 7300	Acceptable	EPA 8270C
6385	2-Methylnaphthalene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6400	2-Methylphenol	µg/kg	2180	9000	230 - 9000	Acceptable	EPA 8270C
6412	3 & 4-Methylphenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5005	Naphthalene	µg/kg	4330	6690	1030 - 7990	Acceptable	EPA 8270C
6460	2-Nitroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6465	3-Nitroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6470	4-Nitroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5015	Nitrobenzene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6490	2-Nitrophenol	µg/kg	3180	5200	233 - 6110	Acceptable	EPA 8270C
6500	4-Nitrophenol	µg/kg	5040	7280	0.00 - 9370	Acceptable	EPA 8270C
6530	N-Nitrosodimethylamine	µg/kg	3570	4730	0.00 - 6190	Acceptable	EPA 8270C
6535	N-Nitrosodiphenylamine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6545	N-Nitroso-di-n-propylamine	µg/kg	1120	1500	102 - 2060	Acceptable	EPA 8270C
6605	Pentachlorophenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6615	Phenanthrene	µg/kg	2950	3650	1600 - 4520	Acceptable	EPA 8270C
6625	Phenol	µg/kg	3230	5750	457 - 6400	Acceptable	EPA 8270C
6665	Pyrene	µg/kg	2260	2850	1030 - 3750	Acceptable	EPA 8270C
5095	Pyridine	µg/kg	< 660	0.00		Acceptable	EPA 8270C
5155	1,2,4-Trichlorobenzene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6835	2,4,5-Trichlorophenol	µg/kg	4420	8390	600 - 9720	Acceptable	EPA 8270C
6840	2,4,6-Trichlorophenol	µg/kg	4950	9400	680 - 10800	Acceptable	EPA 8270C

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP®

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Report Issued: 07/01/04
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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil							
5145	2-Amino-1-methylbenzene (o-toluidine)	µg/kg		0.00			
5625	Benzotrichloride	µg/kg		0.00			
5635	Benzyl chloride	µg/kg		0.00			
6350	bis(2-methoxyethyl)phthalate	µg/kg		0.00			
5810	2-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5815	3-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5820	4-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5885	Diamylphthalate	µg/kg		0.00			
5920	2,4-Dibromophenyl-4-nitrophenylether	µg/kg		0.00			
6010	2,4-Dichlorophenyl-3-methyl-4-nitrophenylether	µg/kg		0.00			
6015	2,3-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6020	2,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6025	2,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6030	2,6-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6035	3,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6040	3,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6055	Dicyclohexylphthalate	µg/kg		0.00			
6505	4-Nitrophenylphenylether	µg/kg		0.00			
6195	Dinonylphthalate	µg/kg		0.00			
6280	Hexachlorocyclohexane	µg/kg		0.00			
6305	Hexyl-2-ethylhexylphthalate	µg/kg		0.00			
6335	Maleic anhydride	µg/kg		0.00			
6590	Pentachlorobenzene	µg/kg		0.00			
6595	Pentachlorohexane	µg/kg		0.00			
6717	Tetrachlorobenzenes	µg/kg		0.00			
6705	1,2,3,4-Tetrachlorobenzene	µg/kg		0.00			
6710	1,2,3,5-Tetrachlorobenzene	µg/kg		0.00			
6715	1,2,4,5-Tetrachlorobenzene	µg/kg		0.00			
6730	2,3,4,5-Tetrachlorophenol	µg/kg		0.00			
6735	2,3,4,6-Tetrachlorophenol	µg/kg		0.00			
6740	2,3,5,6-Tetrachlorophenol	µg/kg		0.00			
5150	1,2,3-Trichlorobenzene	µg/kg		0.00			
6800	1,3,5-Trichlorobenzene	µg/kg		0.00			
6845	2,3,4-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6850	2,3,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6855	2,3,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6860	2,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			

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NVLAP® Lab code: 200386-0

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SOIL-46 Final Complete Report

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Study Dates: 04/26/04 - 06/10/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil (Cont.)							
6865	2,4,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6870	3,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
Pesticides in Soil							
7025	Aldrin	µg/kg		201	85.8 - 298		
7110	alpha-BHC	µg/kg		137	44.8 - 197		
7115	beta-BHC	µg/kg		155	26.0 - 224		
7105	delta-BHC	µg/kg		329	87.1 - 461		
7120	gamma-BHC(Lindane)	µg/kg		211	85.4 - 279		
7240	alpha-Chlordane	µg/kg		190	67.9 - 258		
7245	gamma-Chlordane	µg/kg		152	54.6 - 203		
7355	4,4'-DDD	µg/kg		142	58.5 - 206		
7360	4,4'-DDE	µg/kg		151	50.4 - 226		
7365	4,4'-DDT	µg/kg		123	37.2 - 185		
7470	Dieldrin	µg/kg		96.7	43.2 - 133		
7540	Endrin	µg/kg		133	61.6 - 198		
7530	Endrin aldehyde	µg/kg		207	0.00 - 243		
7535	Endrin ketone	µg/kg		232	78.1 - 320		
7510	Endosulfan I	µg/kg		302	94.9 - 381		
7515	Endosulfan II	µg/kg		231	60.2 - 292		
7520	Endosulfan sulfate	µg/kg		205	49.5 - 311		
7685	Heptachlor	µg/kg		196	72.0 - 270		
7690	Heptachlor epoxide	µg/kg		149	53.0 - 209		
7810	Methoxychlor	µg/kg		394	37.0 - 643		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-46 Final Complete Report

Tenkasi Viswanathan
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Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 07/01/04
Study Dates: 04/26/04 - 06/10/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
PAHs in Soil							
5500	Acenaphthene	µg/kg		314	0.00 - 424		
5505	Acenaphthylene	µg/kg	113	214	0.00 - 238	Acceptable	EPA 8310 UV
5555	Anthracene	µg/kg	24.3	90.8	0.00 - 90.8	Acceptable	EPA 8310 UV
5575	Benzo(a)anthracene	µg/kg	48.2	79.7	0.00 - 115	Acceptable	EPA 8310 UV
5585	Benzo(b)fluoranthene	µg/kg	60.2	78.4	0.00 - 133	Acceptable	EPA 8310 UV
5600	Benzo(k)fluoranthene	µg/kg	49.0	74.5	0.00 - 116	Acceptable	EPA 8310 UV
5590	Benzo(g,h,i)perylene	µg/kg	94.5	50.5	0.00 - 88.9	Not Acceptable	EPA 8310 UV
5580	Benzo(a)pyrene	µg/kg	39.2	84.3	0.00 - 123	Acceptable	EPA 8310 UV
5855	Chrysene	µg/kg	47.6	94.6	0.00 - 152	Acceptable	EPA 8310 UV
5895	Dibenz(a,h)anthracene	µg/kg	30.8	44.1	0.00 - 66.0	Acceptable	EPA 8310 UV
6265	Fluoranthene	µg/kg	80.8	53.3	0.00 - 106	Acceptable	EPA 8310 UV
6270	Fluorene	µg/kg	18.7	221	0.00 - 303	Acceptable	EPA 8310 UV
6315	Indeno(1,2,3-cd)pyrene	µg/kg	24.5	83.3	0.00 - 133	Acceptable	EPA 8310 UV
5005	Naphthalene	µg/kg	218	419	0.00 - 534	Acceptable	EPA 8310 UV
6615	Phenanthrene	µg/kg	42.3	89.1	0.00 - 139	Acceptable	EPA 8310 UV
6665	Pyrene	µg/kg	51.7	60.1	0.00 - 98.2	Acceptable	EPA 8310 UV

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES® SOIL-48 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 **EPA ID: KS00902**

Report Issued: 12/22/04
Study Dates: 10/19/04 - 12/03/04

SOIL Study Definitions:

The reported value is the value that the laboratory reported to ERA.

The ERA assigned value for the Organic Proficiency Testing Standards is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation as applicable. The ERA assigned value for the Inorganic Proficiency Testing Standards is equal to the maximum amount of the parameter available in the standard by applicable EPA methodologies. All NELAC parameters not added to a standard are given an assigned Value of "0", per the guidance issued by the NELAC Board of Directors, on December 14, 2000. Non-NELAC parameters not added to a standard may be given an assigned value of less than a minimum verified concentration as determined in the background soil for applicable EPA methodologies.

The acceptance limits are established per the NELAC PT program criteria or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For a further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside of the Acceptance Limits.
No Evaluation	=	Reported Value cannot be evaluated.

The method description is the method the laboratory reported to ERA.

SOIL Study Discussion:

ERA's Proficiency Testing (PT) Study, SOIL-48, has been reviewed by ERA Senior Management and certified compliant with the requirements of the National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program Standards, Chapter 2, July 2003.

Per the requirements of the NELAC Proficiency Testing Program, a full review of all accuracy verification, homogeneity and stability data was completed. All analytical verification data for all analytes in the SOIL-48 standards met the acceptance criteria contained in the NELAC Proficiency Testing Program Standards, Chapter 2, July 2003.

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's review of the data there was one anomaly noted. This anomaly is addressed on the following page.

The Proficiency Testing Study, SOIL-48, report shall not be reproduced except in its entirety and not without the permission of the participating laboratory. The report must not be used by the participating laboratory to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding ERA's Proficiency Testing Study, SOIL-48, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

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Study Discussion - Soil 48: Upper Acceptance Limits

During the review of the Soil-48 data ERA observed several analytes whose NELAC calculated upper acceptance limits were determined to be a value less than the assigned value for the analyte. Per NELAC Chapter 2, Appendix C and the NELAC PT Fields of Testing - RCRA - Solid, the acceptance criteria for soil studies are to be set to ± 3 standard deviations about the mean of the study data. The NELAC Standard Chapter 2 does not address the situation of when the upper acceptance limits are determined to be less than the assigned value. To minimize the possibility of penalizing those laboratories who routinely use methods designed to improve recoveries of these analytes, ERA has set the upper acceptance limits to the assigned value. ERA has communicated this to the NELAC PT Board.

If you have any questions please feel free to call Shawn Kassner, Manger, Proficiency Testing Program, or Jeff Lowry, Director of Proficiency Testing at 1-800-372-0122.

Study Discussion: PCBs in Soil

During the review of the SOIL-48 PCBs in Soil data set, ERA observed a failure rate of 23.0% for Aroclor 1242. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification and stability data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'.

Upon further examination of the PCBs in Soil data set, ERA found that seventeen laboratories misidentified Aroclor 1016 as Aroclor 1242. The failure rate for Aroclor 1242 without the apparent aroclor misidentification is 4.29%. This failure rate is more consistent with ERA's historical failure rate of 4.90%.

If you are one of the laboratories that misidentified Aroclor 1016 and would like technical assistance please call ERA's Organic Chemistry Department at 1-800-372-0122.

ENVIRONMENTAL RESOURCE ASSOCIATES®

Study: **SOIL-48**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method A**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-48 Final Complete Report

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Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/22/04
Study Dates: 10/19/04 - 12/03/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil							
1000	Aluminum	mg/kg	5020	8660	2510 - 12800	Acceptable	EPA 6010B
1005	Antimony	mg/kg	183	194	0.00 - 227	Acceptable	EPA 6010B
1010	Arsenic	mg/kg	91.7	86.1	56.0 - 105	Acceptable	EPA 6010B
1015	Barium	mg/kg	163	159	120 - 192	Acceptable	EPA 6010B
1020	Beryllium	mg/kg	156	155	110 - 179	Acceptable	EPA 6010B
1025	Boron	mg/kg		130	58.5 - 135		
1030	Cadmium	mg/kg	260	264	168 - 302	Acceptable	EPA 6010B
1035	Calcium	mg/kg	4680	4730	3200 - 5480	Acceptable	EPA 6010B
1040	Chromium	mg/kg	64.6	68.0	39.7 - 81.1	Acceptable	EPA 6010B
1050	Cobalt	mg/kg	77.9	81.0	50.9 - 85.3	Acceptable	EPA 6010B
1055	Copper	mg/kg	149	144	101 - 161	Acceptable	EPA 6010B
1070	Iron	mg/kg	11400	15300	1780 - 24800	Acceptable	EPA 6010B
1075	Lead	mg/kg	84.3	84.2	56.7 - 99.1	Acceptable	EPA 6010B
1085	Magnesium	mg/kg	1970	2410	1470 - 2990	Acceptable	EPA 6010B
1090	Manganese	mg/kg	326	315	219 - 385	Acceptable	EPA 6010B
1095	Mercury	mg/kg	3.24	3.66	2.10 - 5.10	Acceptable	EPA 7471A
1100	Molybdenum	mg/kg	71.4	68.1	36.3 - 79.5	Acceptable	EPA 6010B
1105	Nickel	mg/kg	55.5	60.0	36.1 - 64.3	Acceptable	EPA 6010B
1125	Potassium	mg/kg	1970	2300	1390 - 3390	Acceptable	EPA 6010B
1140	Selenium	mg/kg	102	94.9	56.4 - 110	Acceptable	EPA 6010B
1150	Silver	mg/kg	95.0	89.0	49.9 - 110	Acceptable	EPA 6010B
1155	Sodium	mg/kg	473	483	269 - 643	Acceptable	EPA 6010B
1160	Strontium	mg/kg		120	88.4 - 140		
1165	Thallium	mg/kg	179	178	109 - 214	Acceptable	EPA 6010B
1175	Tin	mg/kg		194	107 - 241		
1180	Titanium	mg/kg		320	0.00 - 598		
1185	Vanadium	mg/kg	73.6	82.3	37.5 - 101	Acceptable	EPA 6010B
1190	Zinc	mg/kg	119	126	80.3 - 152	Acceptable	EPA 6010B
Cyanide in Soil							
1635	Cyanide, total	mg/kg	245	449	0.00 - 648	Acceptable	EPA 9010B
1640	Reactive Cyanide	mg/kg	< 1.0	< 25.0	< 25.0	Acceptable	EPA 9010B
Hexavalent Chromium in Soil							
1045	Chromium VI	mg/kg	38.2	155	0.00 - 155	Acceptable	EPA 7196A

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-48 Final Complete Report

Tenkasi Viswanathan
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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/22/04
Study Dates: 10/19/04 - 12/03/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil							
5500	Acenaphthene	µg/kg		5980	1470 - 6710		
5505	Acenaphthylene	µg/kg		0.00			
5545	Aniline	µg/kg		0.00			
5555	Anthracene	µg/kg		0.00			
5595	Benzidine	µg/kg		0.00			
5610	Benzoic acid	µg/kg		0.00			
5575	Benzo(a)anthracene	µg/kg		4380	1420 - 5120		
5585	Benzo(b)fluoranthene	µg/kg		3140	894 - 3730		
5600	Benzo(k)fluoranthene	µg/kg		2580	793 - 3250		
5590	Benzo(g,h,i)perylene	µg/kg		0.00			
5580	Benzo(a)pyrene	µg/kg		0.00			
5630	Benzyl alcohol	µg/kg		0.00			
5660	4-Bromophenyl-phenylether	µg/kg		0.00			
5670	Butylbenzylphthalate	µg/kg		1270	498 - 1600		
5680	Carbazole	µg/kg		0.00			
5745	4-Chloroaniline	µg/kg		0.00			
5760	bis(2-Chloroethoxy)methane	µg/kg		0.00			
5765	bis(2-Chloroethyl)ether	µg/kg		0.00			
5780	bis(2-Chloroisopropyl)ether	µg/kg		6180	720 - 7320		
5700	4-Chloro-3-methylphenol	µg/kg		0.00			
5790	1-Chloronaphthalene	µg/kg		0.00			
5795	2-Chloronaphthalene	µg/kg		0.00			
5800	2-Chlorophenol	µg/kg		1730	96.0 - 1820		
5825	4-Chlorophenyl-phenylether	µg/kg		1170	307 - 1440		
5855	Chrysene	µg/kg		6070	1960 - 7440		
5895	Dibenz(a,h)anthracene	µg/kg		0.00			
5905	Dibenzofuran	µg/kg		1540	445 - 1860		
5925	Di-n-butylphthalate	µg/kg		2340	702 - 3080		
4610	1,2-Dichlorobenzene	µg/kg		1840	203 - 1940		
4615	1,3-Dichlorobenzene	µg/kg		4650	363 - 4650		
4620	1,4-Dichlorobenzene	µg/kg		5250	447 - 5250		
5945	3,3'-Dichlorobenzidine	µg/kg		0.00			
6000	2,4-Dichlorophenol	µg/kg		1500	23.0 - 1640		
6005	2,6-Dichlorophenol	µg/kg		0.00			
6070	Diethylphthalate	µg/kg		5640	1250 - 6870		
6130	2,4-Dimethylphenol	µg/kg		0.00			
6135	Dimethylphthalate	µg/kg		1450	357 - 1800		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-48 Final Complete Report

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Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/22/04
Study Dates: 10/19/04 - 12/03/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)							
6360	4,6-Dinitro-2-methylphenol	µg/kg		0.00			
6175	2,4-Dinitrophenol	µg/kg		0.00			
6185	2,4-Dinitrotoluene	µg/kg		6890	740 - 8840		
6190	2,6-Dinitrotoluene	µg/kg		6230	1370 - 7550		
6200	Di-n-octylphthalate	µg/kg		1490	483 - 1980		
6255	bis(2-Ethylhexyl)phthalate	µg/kg		0.00			
6265	Fluoranthene	µg/kg		1810	629 - 2170		
6270	Fluorene	µg/kg		6680	1800 - 7780		
6275	Hexachlorobenzene	µg/kg		0.00			
4835	Hexachlorobutadiene	µg/kg		1760	192 - 2090		
6285	Hexachlorocyclopentadiene	µg/kg		0.00			
4840	Hexachloroethane	µg/kg		7690	580 - 7690		
6315	Indeno(1,2,3-cd)pyrene	µg/kg		0.00			
6320	Isophorone	µg/kg		5650	767 - 6590		
6385	2-Methylnaphthalene	µg/kg		5890	1220 - 6700		
6400	2-Methylphenol	µg/kg		1580	0.00 - 1580		
6412	3 & 4-Methylphenol	µg/kg		0.00			
5005	Naphthalene	µg/kg		6740	1230 - 7230		
6460	2-Nitroaniline	µg/kg		0.00			
6465	3-Nitroaniline	µg/kg		0.00			
6470	4-Nitroaniline	µg/kg		0.00			
5015	Nitrobenzene	µg/kg		7250	1460 - 7880		
6490	2-Nitrophenol	µg/kg		9140	660 - 9840		
6500	4-Nitrophenol	µg/kg		6020	0.00 - 7280		
6530	N-Nitrosodimethylamine	µg/kg		0.00			
6535	N-Nitrosodiphenylamine	µg/kg		0.00			
6545	N-Nitroso-di-n-propylamine	µg/kg		10300	1110 - 12200		
6605	Pentachlorophenol	µg/kg		2920	0.00 - 3410		
6615	Phenanthrene	µg/kg		0.00			
6625	Phenol	µg/kg		1060	66.0 - 1170		
6665	Pyrene	µg/kg		3920	1430 - 4310		
5095	Pyridine	µg/kg		0.00			
5155	1,2,4-Trichlorobenzene	µg/kg		0.00			
6835	2,4,5-Trichlorophenol	µg/kg		1700	68.0 - 1840		
6840	2,4,6-Trichlorophenol	µg/kg		1230	75.0 - 1230		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-48 Final Complete Report

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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/22/04
Study Dates: 10/19/04 - 12/03/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil							
5145	2-Amino-1-methylbenzene (o-toluidine)	µg/kg		0.00			
5625	Benzotrichloride	µg/kg		0.00			
5635	Benzyl chloride	µg/kg		0.00			
6350	bis(2-methoxyethyl)phthalate	µg/kg		0.00			
5810	2-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5815	3-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5820	4-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5885	Diamylphthalate	µg/kg		0.00			
5920	2,4-Dibromophenyl-4-nitrophenylether	µg/kg		0.00			
6010	2,4-Dichlorophenyl-3-methyl-4-nitrophenylether	µg/kg		0.00			
6015	2,3-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6020	2,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6025	2,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6030	2,6-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6035	3,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6040	3,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6055	Dicyclohexylphthalate	µg/kg		0.00			
6505	4-Nitrophenylphenylether	µg/kg		0.00			
6195	Dinonylphthalate	µg/kg		0.00			
6280	Hexachlorocyclohexane	µg/kg		0.00			
6305	Hexyl-2-ethylhexylphthalate	µg/kg		0.00			
6335	Maleic anhydride	µg/kg		0.00			
6590	Pentachlorobenzene	µg/kg		0.00			
6595	Pentachlorohexane	µg/kg		0.00			
6717	Tetrachlorobenzenes	µg/kg		0.00			
6705	1,2,3,4-Tetrachlorobenzene	µg/kg		0.00			
6710	1,2,3,5-Tetrachlorobenzene	µg/kg		0.00			
6715	1,2,4,5-Tetrachlorobenzene	µg/kg		0.00			
6730	2,3,4,5-Tetrachlorophenol	µg/kg		0.00			
6735	2,3,4,6-Tetrachlorophenol	µg/kg		0.00			
6740	2,3,5,6-Tetrachlorophenol	µg/kg		0.00			
5150	1,2,3-Trichlorobenzene	µg/kg		0.00			
6800	1,3,5-Trichlorobenzene	µg/kg		0.00			
6845	2,3,4-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6850	2,3,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6855	2,3,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6860	2,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-48 Final Complete Report

Tenkasi Viswanathan
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Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/22/04
Study Dates: 10/19/04 - 12/03/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil (Cont.)							
6865	2,4,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6870	3,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
Pesticides in Soil							
7025	Aldrin	µg/kg	332	453	144 - 616	Acceptable	EPA 8081A
7110	alpha-BHC	µg/kg	324	456	101 - 661	Acceptable	EPA 8081A
7115	beta-BHC	µg/kg	262	412	62.9 - 589	Acceptable	EPA 8081A
7105	delta-BHC	µg/kg	265	414	90.7 - 589	Acceptable	EPA 8081A
7120	gamma-BHC(Lindane)	µg/kg	322	458	111 - 643	Acceptable	EPA 8081A
7240	alpha-Chlordane	µg/kg	129	197	64.2 - 266	Acceptable	EPA 8081A
7245	gamma-Chlordane	µg/kg	94.6	148	43.5 - 203	Acceptable	EPA 8081A
7355	4,4'-DDD	µg/kg	264	411	122 - 596	Acceptable	EPA 8081A
7360	4,4'-DDE	µg/kg	268	413	158 - 560	Acceptable	EPA 8081A
7365	4,4'-DDT	µg/kg	207	385	91.9 - 564	Acceptable	EPA 8081A
7470	Dieldrin	µg/kg	130	212	63.2 - 307	Acceptable	EPA 8081A
7540	Endrin	µg/kg	95.1	132	39.5 - 205	Acceptable	EPA 8081A
7530	Endrin aldehyde	µg/kg	108	210	6.90 - 305	Acceptable	EPA 8081A
7535	Endrin ketone	µg/kg	114	180	57.2 - 259	Acceptable	EPA 8081A
7510	Endosulfan I	µg/kg	50.6	77.6	18.6 - 116	Acceptable	EPA 8081A
7515	Endosulfan II	µg/kg	67.4	106	21.6 - 149	Acceptable	EPA 8081A
7520	Endosulfan sulfate	µg/kg	87.1	139	26.7 - 207	Acceptable	EPA 8081A
7685	Heptachlor	µg/kg	304	419	96.6 - 611	Acceptable	EPA 8081A
7690	Heptachlor epoxide	µg/kg	301	414	146 - 556	Acceptable	EPA 8081A
7810	Methoxychlor	µg/kg	205	254	54.8 - 387	Acceptable	EPA 8081A
Chlordane in Soil							
7250	Chlordane, Technical	µg/kg	116	148	28.3 - 208	Acceptable	EPA 8081A
Toxaphene in Soil							
8250	Toxaphene	µg/kg	130	166	0.00 - 250	Acceptable	EPA 8081A
Volatiles in Soil							
4315	Acetone	µg/kg	62.2	197	0.00 - 365	Acceptable	EPA 8260B
4320	Acetonitrile	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4325	Acrolein	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4375	Benzene	µg/kg	26.1	24.0	14.9 - 32.6	Acceptable	EPA 8260B
4395	Bromodichloromethane	µg/kg	196	185	121 - 257	Acceptable	EPA 8260B
4400	Bromoform	µg/kg	201	199	94.3 - 304	Acceptable	EPA 8260B

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-48 Final Complete Report

Tenkasi Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/22/04
Study Dates: 10/19/04 - 12/03/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)							
4950	Bromomethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4410	2-Butanone (MEK)	µg/kg	39.2	150	0.00 - 235	Acceptable	EPA 8260B
4450	Carbon disulfide	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4455	Carbon tetrachloride	µg/kg	211	197	93.0 - 297	Acceptable	EPA 8260B
4475	Chlorobenzene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4575	Chlorodibromomethane	µg/kg	198	192	121 - 263	Acceptable	EPA 8260B
4485	Chloroethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4500	2-Chloroethylvinylether	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4505	Chloroform	µg/kg	205	198	113 - 275	Acceptable	EPA 8260B
4960	Chloromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4570	1,2-Dibromo-3-chloropropane (DBCP)	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4585	1,2-Dibromoethane (EDB)	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4595	Dibromomethane	µg/kg	30.6	29.2	17.7 - 40.5	Acceptable	EPA 8260B
4610	1,2-Dichlorobenzene	µg/kg	206	195	57.2 - 295	Acceptable	EPA 8260B
4615	1,3-Dichlorobenzene	µg/kg	211	193	61.5 - 293	Acceptable	EPA 8260B
4620	1,4-Dichlorobenzene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4625	Dichlorodifluoromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4630	1,1-Dichloroethane	µg/kg	21.0	20.4	12.3 - 28.8	Acceptable	EPA 8260B
4635	1,2-Dichloroethane	µg/kg	22.0	20.6	13.0 - 28.6	Acceptable	EPA 8260B
4640	1,1-Dichloroethylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4645	cis-1,2-Dichloroethylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4700	trans-1,2-Dichloroethylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4655	1,2-Dichloropropane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4680	cis-1,3-Dichloropropylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4685	trans-1,3-Dichloropropylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4765	Ethylbenzene	µg/kg	173	157	84.3 - 216	Acceptable	EPA 8260B
4860	2-Hexanone	µg/kg	173	142	73.6 - 270	Acceptable	EPA 8260B
4975	Methylene chloride	µg/kg	28.3	23.0	7.99 - 37.8	Acceptable	EPA 8260B
4995	4-Methyl-2-pentanone (MIBK)	µg/kg	130	128	54.9 - 179	Acceptable	EPA 8260B
5100	Styrene	µg/kg	168	169	75.4 - 245	Acceptable	EPA 8260B
5105	1,1,1,2-Tetrachloroethane	µg/kg	103	101	59.6 - 137	Acceptable	EPA 8260B
5110	1,1,2,2-Tetrachloroethane	µg/kg	20.8	22.9	11.5 - 34.3	Acceptable	EPA 8260B
5115	Tetrachloroethylene	µg/kg	81.5	80.7	29.1 - 120	Acceptable	EPA 8260B
5140	Toluene	µg/kg	40.5	40.4	24.4 - 55.6	Acceptable	EPA 8260B
5160	1,1,1-Trichloroethane	µg/kg	207	197	104 - 286	Acceptable	EPA 8260B
5165	1,1,2-Trichloroethane	µg/kg	23.5	23.5	15.2 - 31.4	Acceptable	EPA 8260B
5170	Trichloroethylene	µg/kg	194	193	98.0 - 272	Acceptable	EPA 8260B

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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SOIL-48 Final Complete Report

Tenkasi Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/22/04
Study Dates: 10/19/04 - 12/03/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)							
5175	Trichlorofluoromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5180	1,2,3-Trichloropropane	µg/kg	73.4	82.0	21.7 - 133	Acceptable	EPA 8260B
5225	Vinyl acetate	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5235	Vinyl chloride	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5260	Total, Xylenes	µg/kg	50.4	45.7	16.0 - 78.4	Acceptable	EPA 8260B
Herbicides in Soil							
8545	2,4-D	µg/kg	3.84	103	0.00 - 182	Acceptable	EPA 8151A
8555	Dalapon	µg/kg	< 5.0	0.00		Acceptable	EPA 8151A
8560	2,4-DB	µg/kg	182	2430	0.00 - 3670	Acceptable	EPA 8151A
8595	Dicamba	µg/kg	3.30	493	0.00 - 683	Acceptable	EPA 8151A
8620	Dinoseb	µg/kg	1170	2350	0.00 - 3000	Acceptable	EPA 8151A
7780	MCPP	µg/kg	< 250	0.00		Acceptable	EPA 8151A
6605	Pentachlorophenol	µg/kg	901	2460	0.00 - 3470	Acceptable	EPA 8151A
8655	2,4,5-T	µg/kg	2.39	109	0.00 - 180	Acceptable	EPA 8151A
8650	2,4,5-TP (Silvex)	µg/kg	13.1	479	0.00 - 713	Acceptable	EPA 8151A
PCBs in Soil							
8880	Aroclor 1016	mg/kg	11.8	26.2	0.00 - 33.1	Acceptable	EPA 8082A
8885	Aroclor 1221	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8890	Aroclor 1232	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8895	Aroclor 1242	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8900	Aroclor 1248	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8905	Aroclor 1254	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8910	Aroclor 1260	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Labcode 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-48 Final Complete Report

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QA Director
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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/22/04
Study Dates: 10/19/04 - 12/03/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
PAHs in Soil							
5500	Acenaphthene	µg/kg	295	476	62.8 - 539	Acceptable	EPA 8270 SIM
5505	Acenaphthylene	µg/kg	177	326	27.9 - 326	Acceptable	EPA 8270 SIM
5555	Anthracene	µg/kg	31.0	73.8	8.59 - 73.8	Acceptable	EPA 8270 SIM
5575	Benzo(a)anthracene	µg/kg	39.3	53.1	18.9 - 61.9	Acceptable	EPA 8270 SIM
5585	Benzo(b)fluoranthene	µg/kg	38.0	47.8	22.6 - 58.6	Acceptable	EPA 8270 SIM
5600	Benzo(k)fluoranthene	µg/kg	38.8	54.8	18.4 - 65.8	Acceptable	EPA 8270 SIM
5590	Benzo(g,h,i)perylene	µg/kg	50.0	66.7	16.3 - 86.5	Acceptable	EPA 8270 SIM
5580	Benzo(a)pyrene	µg/kg	34.3	54.0	10.0 - 66.8	Acceptable	EPA 8270 SIM
5855	Chrysene	µg/kg	66.2	96.6	43.7 - 108	Acceptable	EPA 8270 SIM
5895	Dibenz(a,h)anthracene	µg/kg	31.4	48.5	14.7 - 53.1	Acceptable	EPA 8270 SIM
6265	Fluoranthene	µg/kg	64.8	86.3	38.8 - 101	Acceptable	EPA 8270 SIM
6270	Fluorene	µg/kg	329	480	94.9 - 525	Acceptable	EPA 8270 SIM
6315	Indeno(1,2,3-cd)pyrene	µg/kg	41.4	49.4	14.3 - 62.6	Acceptable	EPA 8270 SIM
5005	Naphthalene	µg/kg	295	476	0.00 - 600	Acceptable	EPA 8270 SIM
6615	Phenanthrene	µg/kg	40.0	59.4	23.0 - 69.8	Acceptable	EPA 8270 SIM
6665	Pyrene	µg/kg	30.9	45.8	16.0 - 60.4	Acceptable	EPA 8270 SIM
Nitroaromatics & Nitramines in Soil							
9306	4-Amino-2,6-dinitrotoluene	µg/kg	< 100	0.00		Acceptable	EPA 8330
9303	2-Amino-4,6-dinitrotoluene	µg/kg	< 100	0.00		Acceptable	EPA 8330
6160	1,3-Dinitrobenzene	µg/kg	15300	15100	8080 - 20300	Acceptable	EPA 8330
6185	2,4-Dinitrotoluene	µg/kg	< 100	0.00		Acceptable	EPA 8330
6190	2,6-Dinitrotoluene	µg/kg	9960	9340	3790 - 13400	Acceptable	EPA 8330
9522	HMX	µg/kg	2550	2610	1740 - 3660	Acceptable	EPA 8330
5015	Nitrobenzene	µg/kg	26200	24900	12500 - 35100	Acceptable	EPA 8330
9507	2-Nitrotoluene	µg/kg	16300	14900	7400 - 20000	Acceptable	EPA 8330
9510	3-Nitrotoluene	µg/kg	< 100	0.00		Acceptable	EPA 8330
9513	4-Nitrotoluene	µg/kg	< 100	0.00		Acceptable	EPA 8330
9432	RDX	µg/kg	12900	13200	8540 - 16500	Acceptable	EPA 8330
6415	Tetryl	µg/kg	< 100	0.00		Acceptable	EPA 8330
6885	1,3,5-Trinitrobenzene	µg/kg	< 100	0.00		Acceptable	EPA 8330
9651	2,4,6-Trinitrotoluene	µg/kg	8850	9330	3570 - 11800	Acceptable	EPA 8330
Ignitability							
1780	Ignitability (Flashpoint)	deg. F	118	108	98.7 - 125	Acceptable	EPA 1010
Corrosivity (pH)							
1625	Corrosivity (pH)	S.U.	10.21	10.4	9.92 - 10.7	Acceptable	EPA 9045C

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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Study: **SOIL-48**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method B**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-48 Final Complete Report

Tenkasi Viswanathan
QA Director
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Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/22/04
Study Dates: 10/19/04 - 12/03/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil							
1000	Aluminum	mg/kg	4590	8660	2510 - 12800	Acceptable	EPA 6020
1005	Antimony	mg/kg	156	194	0.00 - 227	Acceptable	EPA 6020
1010	Arsenic	mg/kg	82.7	86.1	56.0 - 105	Acceptable	EPA 6020
1015	Barium	mg/kg	141	159	120 - 192	Acceptable	EPA 6020
1020	Beryllium	mg/kg	142	155	110 - 179	Acceptable	EPA 6020
1025	Boron	mg/kg	84.8	130	58.5 - 135	Acceptable	EPA 6020
1030	Cadmium	mg/kg	236	264	168 - 302	Acceptable	EPA 6020
1035	Calcium	mg/kg	4290	4730	3200 - 5480	Acceptable	EPA 6020
1040	Chromium	mg/kg	57.4	68.0	39.7 - 81.1	Acceptable	EPA 6020
1050	Cobalt	mg/kg	68.7	81.0	50.9 - 85.3	Acceptable	EPA 6020
1055	Copper	mg/kg	130	144	101 - 161	Acceptable	EPA 6020
1070	Iron	mg/kg	10200	15300	1780 - 24800	Acceptable	EPA 6020
1075	Lead	mg/kg	74.7	84.2	56.7 - 99.1	Acceptable	EPA 6020
1085	Magnesium	mg/kg	1750	2410	1470 - 2990	Acceptable	EPA 6020
1090	Manganese	mg/kg	292	315	219 - 385	Acceptable	EPA 6020
1095	Mercury	mg/kg	3.15	3.66	2.10 - 5.10	Acceptable	EPA 6020
1100	Molybdenum	mg/kg	62.1	68.1	36.3 - 79.5	Acceptable	EPA 6020
1105	Nickel	mg/kg	50.3	60.0	36.1 - 64.3	Acceptable	EPA 6020
1125	Potassium	mg/kg	1820	2300	1390 - 3390	Acceptable	EPA 6020
1140	Selenium	mg/kg	85.8	94.9	56.4 - 110	Acceptable	EPA 6020
1150	Silver	mg/kg	78.5	89.0	49.9 - 110	Acceptable	EPA 6020
1155	Sodium	mg/kg	422	483	269 - 643	Acceptable	EPA 6020
1160	Strontium	mg/kg	106	120	88.4 - 140	Acceptable	EPA 6020
1165	Thallium	mg/kg	159	178	109 - 214	Acceptable	EPA 6020
1175	Tin	mg/kg	167	194	107 - 241	Acceptable	EPA 6020
1180	Titanium	mg/kg	138	320	0.00 - 598	Acceptable	EPA 6020
1185	Vanadium	mg/kg	63.9	82.3	37.5 - 101	Acceptable	EPA 6020
1190	Zinc	mg/kg	107	126	80.3 - 152	Acceptable	EPA 6020

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil							
5500	Acenaphthene	µg/kg	3800	5980	1470 - 6710	Acceptable	EPA 8270C
5505	Acenaphthylene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5545	Aniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5555	Anthracene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5595	Benzidine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5610	Benzoic acid	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5575	Benzo(a)anthracene	µg/kg	3070	4380	1420 - 5120	Acceptable	EPA 8270C
5585	Benzo(b)fluoranthene	µg/kg	2260	3140	894 - 3730	Acceptable	EPA 8270C
5600	Benzo(k)fluoranthene	µg/kg	1640	2580	793 - 3250	Acceptable	EPA 8270C
5590	Benzo(g,h,i)perylene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5580	Benzo(a)pyrene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5630	Benzyl alcohol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5660	4-Bromophenyl-phenylether	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5670	Butylbenzylphthalate	µg/kg	1100	1270	498 - 1600	Acceptable	EPA 8270C
5680	Carbazole	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5745	4-Chloroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5760	bis(2-Chloroethoxy)methane	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5765	bis(2-Chloroethyl)ether	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5780	bis(2-Chloroisopropyl)ether	µg/kg	5160	6180	720 - 7320	Acceptable	EPA 8270C
5700	4-Chloro-3-methylphenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5790	1-Chloronaphthalene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5795	2-Chloronaphthalene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5800	2-Chlorophenol	µg/kg	950	1730	96.0 - 1820	Acceptable	EPA 8270C
5825	4-Chlorophenyl-phenylether	µg/kg	924	1170	307 - 1440	Acceptable	EPA 8270C
5855	Chrysene	µg/kg	3570	6070	1960 - 7440	Acceptable	EPA 8270C
5895	Dibenz(a,h)anthracene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5905	Dibenzofuran	µg/kg	1250	1540	445 - 1860	Acceptable	EPA 8270C
5925	Di-n-butylphthalate	µg/kg	1980	2340	702 - 3080	Acceptable	EPA 8270C
4610	1,2-Dichlorobenzene	µg/kg	1270	1840	203 - 1940	Acceptable	EPA 8270C
4615	1,3-Dichlorobenzene	µg/kg	2760	4650	363 - 4650	Acceptable	EPA 8270C
4620	1,4-Dichlorobenzene	µg/kg	2990	5250	447 - 5250	Acceptable	EPA 8270C
5945	3,3'-Dichlorobenzidine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6000	2,4-Dichlorophenol	µg/kg	745	1500	23.0 - 1640	Acceptable	EPA 8270C
6005	2,6-Dichlorophenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6070	Diethylphthalate	µg/kg	3740	5640	1250 - 6870	Acceptable	EPA 8270C
6130	2,4-Dimethylphenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6135	Dimethylphthalate	µg/kg	1150	1450	357 - 1800	Acceptable	EPA 8270C

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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Report Issued: 12/22/04
Study Dates: 10/19/04 - 12/03/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)							
6360	4,6-Dinitro-2-methylphenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6175	2,4-Dinitrophenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6185	2,4-Dinitrotoluene	µg/kg	4990	6890	740 - 8840	Acceptable	EPA 8270C
6190	2,6-Dinitrotoluene	µg/kg	4550	6230	1370 - 7550	Acceptable	EPA 8270C
6200	Di-n-octylphthalate	µg/kg	1330	1490	483 - 1980	Acceptable	EPA 8270C
6255	bis(2-Ethylhexyl)phthalate	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6265	Fluoranthene	µg/kg	1450	1810	629 - 2170	Acceptable	EPA 8270C
6270	Fluorene	µg/kg	4170	6680	1800 - 7780	Acceptable	EPA 8270C
6275	Hexachlorobenzene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
4835	Hexachlorobutadiene	µg/kg	1300	1760	192 - 2090	Acceptable	EPA 8270C
6285	Hexachlorocyclopentadiene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
4840	Hexachloroethane	µg/kg	4220	7690	580 - 7690	Acceptable	EPA 8270C
6315	Indeno(1,2,3-cd)pyrene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6320	Isophorone	µg/kg	3690	5650	767 - 6590	Acceptable	EPA 8270C
6385	2-Methylnaphthalene	µg/kg	3770	5890	1220 - 6700	Acceptable	EPA 8270C
6400	2-Methylphenol	µg/kg	< 125	1580	0.00 - 1580	Acceptable	EPA 8270C
6412	3 & 4-Methylphenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5005	Naphthalene	µg/kg	3480	6740	1230 - 7230	Acceptable	EPA 8270C
6460	2-Nitroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6465	3-Nitroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6470	4-Nitroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5015	Nitrobenzene	µg/kg	4450	7250	1460 - 7880	Acceptable	EPA 8270C
6490	2-Nitrophenol	µg/kg	4860	9140	660 - 9840	Acceptable	EPA 8270C
6500	4-Nitrophenol	µg/kg	3600	6020	0.00 - 7280	Acceptable	EPA 8270C
6530	N-Nitrosodimethylamine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6535	N-Nitrosodiphenylamine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6545	N-Nitroso-di-n-propylamine	µg/kg	7620	10300	1110 - 12200	Acceptable	EPA 8270C
6605	Pentachlorophenol	µg/kg	1150	2920	0.00 - 3410	Acceptable	EPA 8270C
6615	Phenanthrene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6625	Phenol	µg/kg	643	1060	66.0 - 1170	Acceptable	EPA 8270C
6665	Pyrene	µg/kg	2730	3920	1430 - 4310	Acceptable	EPA 8270C
5095	Pyridine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5155	1,2,4-Trichlorobenzene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6835	2,4,5-Trichlorophenol	µg/kg	793	1700	68.0 - 1840	Acceptable	EPA 8270C
6840	2,4,6-Trichlorophenol	µg/kg	576	1230	75.0 - 1230	Acceptable	EPA 8270C

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Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/22/04
Study Dates: 10/19/04 - 12/03/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil							
5145	2-Amino-1-methylbenzene (o-toluidine)	µg/kg		0.00			
5625	Benzotrichloride	µg/kg		0.00			
5635	Benzyl chloride	µg/kg		0.00			
6350	bis(2-methoxyethyl)phthalate	µg/kg		0.00			
5810	2-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5815	3-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5820	4-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5885	Diamylphthalate	µg/kg		0.00			
5920	2,4-Dibromophenyl-4-nitrophenylether	µg/kg		0.00			
6010	2,4-Dichlorophenyl-3-methyl-4-nitrophenylether	µg/kg		0.00			
6015	2,3-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6020	2,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6025	2,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6030	2,6-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6035	3,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6040	3,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6055	Dicyclohexylphthalate	µg/kg		0.00			
6505	4-Nitrophenylphenylether	µg/kg		0.00			
6195	Dinonylphthalate	µg/kg		0.00			
6280	Hexachlorocyclohexane	µg/kg		0.00			
6305	Hexyl-2-ethylhexylphthalate	µg/kg		0.00			
6335	Maleic anhydride	µg/kg		0.00			
6590	Pentachlorobenzene	µg/kg		0.00			
6595	Pentachlorohexane	µg/kg		0.00			
6717	Tetrachlorobenzenes	µg/kg		0.00			
6705	1,2,3,4-Tetrachlorobenzene	µg/kg		0.00			
6710	1,2,3,5-Tetrachlorobenzene	µg/kg		0.00			
6715	1,2,4,5-Tetrachlorobenzene	µg/kg		0.00			
6730	2,3,4,5-Tetrachlorophenol	µg/kg		0.00			
6735	2,3,4,6-Tetrachlorophenol	µg/kg		0.00			
6740	2,3,5,6-Tetrachlorophenol	µg/kg		0.00			
5150	1,2,3-Trichlorobenzene	µg/kg		0.00			
6800	1,3,5-Trichlorobenzene	µg/kg		0.00			
6845	2,3,4-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6850	2,3,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6855	2,3,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6860	2,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-48 Final Complete Report

Tenkasi Viswanathan
QA Director
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15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil (Cont.)							
6865	2,4,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6870	3,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
Pesticides in Soil							
7025	Aldrin	µg/kg		453	144 - 616		
7110	alpha-BHC	µg/kg		456	101 - 661		
7115	beta-BHC	µg/kg		412	62.9 - 589		
7105	delta-BHC	µg/kg		414	90.7 - 589		
7120	gamma-BHC(Lindane)	µg/kg		458	111 - 643		
7240	alpha-Chlordane	µg/kg		197	64.2 - 266		
7245	gamma-Chlordane	µg/kg		148	43.5 - 203		
7355	4,4'-DDD	µg/kg		411	122 - 596		
7360	4,4'-DDE	µg/kg		413	158 - 560		
7365	4,4'-DDT	µg/kg		385	91.9 - 564		
7470	Dieldrin	µg/kg		212	63.2 - 307		
7540	Endrin	µg/kg		132	39.5 - 205		
7530	Endrin aldehyde	µg/kg		210	6.90 - 305		
7535	Endrin ketone	µg/kg		180	57.2 - 259		
7510	Endosulfan I	µg/kg		77.6	18.6 - 116		
7515	Endosulfan II	µg/kg		106	21.6 - 149		
7520	Endosulfan sulfate	µg/kg		139	26.7 - 207		
7685	Heptachlor	µg/kg		419	96.6 - 611		
7690	Heptachlor epoxide	µg/kg		414	146 - 556		
7810	Methoxychlor	µg/kg		254	54.8 - 387		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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SOIL-48 Final Complete Report

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
PAHs in Soil							
5500	Acenaphthene	µg/kg	208	476	62.8 - 539	Acceptable	EPA 8310 UV
5505	Acenaphthylene	µg/kg	123	326	27.9 - 326	Acceptable	EPA 8310 UV
5555	Anthracene	µg/kg	28.9	73.8	8.59 - 73.8	Acceptable	EPA 8310 UV
5575	Benzo(a)anthracene	µg/kg	32.2	53.1	18.9 - 61.9	Acceptable	EPA 8310 UV
5585	Benzo(b)fluoranthene	µg/kg	41.4	47.8	22.6 - 58.6	Acceptable	EPA 8310 UV
5600	Benzo(k)fluoranthene	µg/kg	38.9	54.8	18.4 - 65.8	Acceptable	EPA 8310 UV
5590	Benzo(g,h,i)perylene	µg/kg	42.5	66.7	16.3 - 86.5	Acceptable	EPA 8310 UV
5580	Benzo(a)pyrene	µg/kg	28.5	54.0	10.0 - 66.8	Acceptable	EPA 8310 UV
5855	Chrysene	µg/kg	65.1	96.6	43.7 - 108	Acceptable	EPA 8310 UV
5895	Dibenz(a,h)anthracene	µg/kg	35.5	48.5	14.7 - 53.1	Acceptable	EPA 8310 UV
6265	Fluoranthene	µg/kg	63.3	86.3	38.8 - 101	Acceptable	EPA 8310 UV
6270	Fluorene	µg/kg	240	480	94.9 - 525	Acceptable	EPA 8310 UV
6315	Indeno(1,2,3-cd)pyrene	µg/kg	36.2	49.4	14.3 - 62.6	Acceptable	EPA 8310 UV
5005	Naphthalene	µg/kg	165	476	0.00 - 600	Acceptable	EPA 8310 UV
6615	Phenanthrene	µg/kg	38.2	59.4	23.0 - 69.8	Acceptable	EPA 8310 UV
6665	Pyrene	µg/kg	32.8	45.8	16.0 - 60.4	Acceptable	EPA 8310 UV

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES® SOIL-50 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 **EPA ID: KS00902**

Report Issued: 06/29/05
Study Dates: 04/25/05 - 06/09/05

SOIL Study Definitions:

The reported value is the value that the laboratory reported to ERA.

The ERA assigned value for the Organic Proficiency Testing Standards is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation as applicable. The ERA assigned value for the Inorganic Proficiency Testing Standards is equal to the maximum amount of the parameter available in the standard by applicable EPA methodologies. All NELAC parameters not added to a standard are given an assigned Value of "0", per the guidance issued by the NELAC Board of Directors, on December 14, 2000. Non-NELAC parameters not added to a standard may be given an assigned value of less than a minimum verified concentration as determined in the background soil for applicable EPA methodologies.

The acceptance limits are established per the NELAC PT program criteria or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For a further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable = Reported Value falls within the Acceptance Limits.

Not Acceptable = Reported Value falls outside of the Acceptance Limits.

No Evaluation = Reported Value cannot be evaluated.

The method description is the method the laboratory reported to ERA.

Any Performance Evaluation left blank indicates results were evaluated as 'Not Reported'.

SOIL Study Discussion:

ERA's Proficiency Testing (PT) Study, SOIL-50, has been reviewed by ERA Senior Management and certified compliant with the requirements of the National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program Standards, Chapter 2, July 2003.

Per the requirements of the NELAC Proficiency Testing Program, a full review of all accuracy verification, homogeneity and stability data was completed. All analytical verification data for all analytes in the SOIL-50 standards met the acceptance criteria contained in the NELAC Proficiency Testing Program Standards, Chapter 2, July 2003.

During the review of the SOIL-50 data ERA observed multiple analytes whose NELAC calculated upper acceptance limits were determined to be a value less than the assigned value for the analyte. Per the NELAC Standards, Chapter 2, Appendix C, and the NELAC PT Fields of Testing - RCRA - Solid, the acceptance criteria for soil studies are to be set to ± 3 standard deviations about the mean of the study data. The NELAC Standards, Chapter 2, do not address the situation when the upper acceptance limit is determined to be less than the assigned value. To minimize the possibility of penalizing those laboratories who routinely use methods designed to improve recoveries of these analytes, ERA has set the upper acceptance limits to the assigned value. ERA has communicated this to the NELAC PT Board.

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's review of the data there were no anomalies noted.

The Proficiency Testing Study, SOIL-50, report shall not be reproduced except in its entirety and not without the permission of the participating laboratory. The report must not be used by the participating laboratory to claim product endorsement by NVLAP or any agency of the U. S. government.

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SOIL-50 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 **EPA ID: KS00902**

Report Issued: 06/29/05
Study Dates: 04/25/05 - 06/09/05

SOIL Study Definitions:

SOIL Study Discussion:

If you have any questions regarding ERA's Proficiency Testing Study, SOIL-50, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

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ENVIRONMENTAL RESOURCE ASSOCIATES®

Study: **SOIL-50**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method A**

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-50 Final Complete Report

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Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/29/05
Study Dates: 04/25/05 - 06/09/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil							
1000	Aluminum	mg/kg	4030	8010	2820 - 11500	Acceptable	EPA 6010B
1005	Antimony	mg/kg	145	171	0.00 - 171	Acceptable	EPA 6010B
1010	Arsenic	mg/kg	195	187	129 - 223	Acceptable	EPA 6010B
1015	Barium	mg/kg	380	372	275 - 443	Acceptable	EPA 6010B
1020	Beryllium	mg/kg	123	126	90.2 - 148	Acceptable	EPA 6010B
1025	Boron	mg/kg	105	141	67.4 - 159	Acceptable	EPA 6010B
1030	Cadmium	mg/kg	61.6	61.5	41.8 - 69.2	Acceptable	EPA 6010B
1035	Calcium	mg/kg	4230	4190	2850 - 4990	Acceptable	EPA 6010B
1040	Chromium	mg/kg	123	122	82.7 - 143	Acceptable	EPA 6010B
1050	Cobalt	mg/kg	147	150	94.2 - 164	Acceptable	EPA 6010B
1055	Copper	mg/kg	86.0	85.6	60.3 - 99.6	Acceptable	EPA 6010B
1070	Iron	mg/kg	12050	15300	2820 - 24200	Acceptable	EPA 6010B
1075	Lead	mg/kg	136	133	93.5 - 157	Acceptable	EPA 6010B
1085	Magnesium	mg/kg	2390	2840	1860 - 3510	Acceptable	EPA 6010B
1090	Manganese	mg/kg	278	267	188 - 332	Acceptable	EPA 6010B
1095	Mercury	mg/kg	2.76	2.39	1.43 - 3.53	Acceptable	EPA 7471A
1100	Molybdenum	mg/kg	139	141	90.7 - 157	Acceptable	EPA 6010B
1105	Nickel	mg/kg	189	186	124 - 206	Acceptable	EPA 6010B
1125	Potassium	mg/kg	2450	2820	1800 - 4050	Acceptable	EPA 6010B
1140	Selenium	mg/kg	139	127	83.6 - 148	Acceptable	EPA 6010B
1150	Silver	mg/kg	76.7	70.7	47.0 - 84.8	Acceptable	EPA 6010B
1155	Sodium	mg/kg	483	509	323 - 631	Acceptable	EPA 6010B
1160	Strontium	mg/kg	99.3	113	75.4 - 137	Acceptable	EPA 6010B
1165	Thallium	mg/kg	114	113	71.1 - 133	Acceptable	EPA 6010B
1175	Tin	mg/kg	135	153	89.1 - 187	Acceptable	EPA 6010B
1180	Titanium	mg/kg	160	320	0.00 - 601	Acceptable	EPA 6010B
1185	Vanadium	mg/kg	86.2	91.0	48.2 - 110	Acceptable	EPA 6010B
1190	Zinc	mg/kg	178	181	122 - 214	Acceptable	EPA 6010B
Cyanide in Soil							
1635	Cyanide, total	mg/kg	121	144	0.00 - 230	Acceptable	EPA 9010B
1640	Reactive Cyanide	mg/kg	< 25	< 25.0	< 25.0	Acceptable	EPA 9010B
Hexavalent Chromium in Soil							
1045	Chromium VI	mg/kg	143	263	81.4 - 269	Acceptable	EPA 7196A

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-50 Final Complete Report

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Study Dates: 04/25/05 - 06/09/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil							
5500	Acenaphthene	µg/kg	2980	5190	1260 - 5700	Acceptable	EPA 8270C
5505	Acenaphthylene	µg/kg	778	2020	113 - 2020	Acceptable	EPA 8270C
5545	Aniline	µg/kg		0.00			
5555	Anthracene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5595	Benzidine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5610	Benzoic acid	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5575	Benzo(a)anthracene	µg/kg	2630	3530	1330 - 4110	Acceptable	EPA 8270C
5585	Benzo(b)fluoranthene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5600	Benzo(k)fluoranthene	µg/kg	1060	1330	473 - 1790	Acceptable	EPA 8270C
5590	Benzo(g,h,i)perylene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5580	Benzo(a)pyrene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5630	Benzyl alcohol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5660	4-Bromophenyl-phenylether	µg/kg	7690	12000	3630 - 15300	Acceptable	EPA 8270C
5670	Butylbenzylphthalate	µg/kg	5680	8020	1670 - 11500	Acceptable	EPA 8270C
5680	Carbazole	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5745	4-Chloroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5760	bis(2-Chloroethoxy)methane	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5765	bis(2-Chloroethyl)ether	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5780	bis(2-Chloroisopropyl)ether	µg/kg	3510	4260	741 - 5200	Acceptable	EPA 8270C
5700	4-Chloro-3-methylphenol	µg/kg	4730	9160	1790 - 9830	Acceptable	EPA 8270C
5790	1-Chloronaphthalene	µg/kg		0.00			
5795	2-Chloronaphthalene	µg/kg	4510	9280	1830 - 11100	Acceptable	EPA 8270C
5800	2-Chlorophenol	µg/kg	2380	4050	502 - 4260	Acceptable	EPA 8270C
5825	4-Chlorophenyl-phenylether	µg/kg	7660	10400	3090 - 12900	Acceptable	EPA 8270C
5855	Chrysene	µg/kg	1260	1270	527 - 1730	Acceptable	EPA 8270C
5895	Dibenz(a,h)anthracene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5905	Dibenzofuran	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5925	Di-n-butylphthalate	µg/kg	2120	3930	877 - 5320	Acceptable	EPA 8270C
4610	1,2-Dichlorobenzene	µg/kg	3860	8660	20.0 - 10200	Acceptable	EPA 8270C
4615	1,3-Dichlorobenzene	µg/kg	4270	10800	0.00 - 12400	Acceptable	EPA 8270C
4620	1,4-Dichlorobenzene	µg/kg	2840	5760	200 - 6500	Acceptable	EPA 8270C
5945	3,3'-Dichlorobenzidine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6000	2,4-Dichlorophenol	µg/kg	2950	5650	766 - 6090	Acceptable	EPA 8270C
6005	2,6-Dichlorophenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6070	Diethylphthalate	µg/kg	3380	6000	590 - 8150	Acceptable	EPA 8270C
6130	2,4-Dimethylphenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6135	Dimethylphthalate	µg/kg	2750	3420	767 - 4310	Acceptable	EPA 8270C

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-50 Final Complete Report

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)							
6360	4,6-Dinitro-2-methylphenol	µg/kg	3880	6340	0.00 - 8320	Acceptable	EPA 8270C
6175	2,4-Dinitrophenol	µg/kg	1650	5700	0.00 - 5700	Acceptable	EPA 8270C
6185	2,4-Dinitrotoluene	µg/kg	4630	5760	820 - 8020	Acceptable	EPA 8270C
6190	2,6-Dinitrotoluene	µg/kg	5750	7300	1960 - 9220	Acceptable	EPA 8270C
6200	Di-n-octylphthalate	µg/kg	3640	9630	1090 - 15100	Acceptable	EPA 8270C
6255	bis(2-Ethylhexyl)phthalate	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6265	Fluoranthene	µg/kg	2840	6770	2270 - 8750	Acceptable	EPA 8270C
6270	Fluorene	µg/kg	2540	4240	1220 - 5040	Acceptable	EPA 8270C
6275	Hexachlorobenzene	µg/kg	6380	8080	2840 - 9920	Acceptable	EPA 8270C
4835	Hexachlorobutadiene	µg/kg	1530	2300	449 - 2650	Acceptable	EPA 8270C
6285	Hexachlorocyclopentadiene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
4840	Hexachloroethane	µg/kg	3510	7200	200 - 8000	Acceptable	EPA 8270C
6315	Indeno(1,2,3-cd)pyrene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6320	Isophorone	µg/kg	2420	3160	331 - 4070	Acceptable	EPA 8270C
6385	2-Methylnaphthalene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6400	2-Methylphenol	µg/kg	1750	5830	0.00 - 5830	Acceptable	EPA 8270C
6412	3 & 4-Methylphenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5005	Naphthalene	µg/kg	1310	2110	370 - 2530	Acceptable	EPA 8270C
6460	2-Nitroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6465	3-Nitroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6470	4-Nitroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5015	Nitrobenzene	µg/kg	4820	9270	1060 - 11300	Acceptable	EPA 8270C
6490	2-Nitrophenol	µg/kg	3840	7050	230 - 8030	Acceptable	EPA 8270C
6500	4-Nitrophenol	µg/kg	1900	2300	0.00 - 3470	Acceptable	EPA 8270C
6530	N-Nitrosodimethylamine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6535	N-Nitrosodiphenylamine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6545	N-Nitroso-di-n-propylamine	µg/kg	6370	8750	410 - 11200	Acceptable	EPA 8270C
6605	Pentachlorophenol	µg/kg	878	1690	0.00 - 2130	Acceptable	EPA 8270C
6615	Phenanthrene	µg/kg	1340	1890	727 - 2350	Acceptable	EPA 8270C
6625	Phenol	µg/kg	6460	12600	910 - 13900	Acceptable	EPA 8270C
6665	Pyrene	µg/kg	3110	4860	1600 - 5880	Acceptable	EPA 8270C
5095	Pyridine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5155	1,2,4-Trichlorobenzene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6835	2,4,5-Trichlorophenol	µg/kg	2380	4230	458 - 4800	Acceptable	EPA 8270C
6840	2,4,6-Trichlorophenol	µg/kg	3040	5010	840 - 5700	Acceptable	EPA 8270C

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil							
5145	2-Amino-1-methylbenzene (o-toluidine)	µg/kg		0.00			
5625	Benzotrichloride	µg/kg		0.00			
5635	Benzyl chloride	µg/kg		0.00			
6350	bis(2-methoxyethyl)phthalate	µg/kg		0.00			
5810	2-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5815	3-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5820	4-Chlorophenyl-4-nitrophenylether	µg/kg		0.00			
5885	Diamylphthalate	µg/kg		0.00			
5920	2,4-Dibromophenyl-4-nitrophenylether	µg/kg		0.00			
6010	2,4-Dichlorophenyl-3-methyl-4-nitrophenylether	µg/kg		0.00			
6015	2,3-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6020	2,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6025	2,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6030	2,6-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6035	3,4-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6040	3,5-Dichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6055	Dicyclohexylphthalate	µg/kg		0.00			
6505	4-Nitrophenylphenylether	µg/kg		0.00			
6195	Dinonylphthalate	µg/kg		0.00			
6280	Hexachlorocyclohexane	µg/kg		0.00			
6305	Hexyl-2-ethylhexylphthalate	µg/kg		0.00			
6335	Maleic anhydride	µg/kg		0.00			
6590	Pentachlorobenzene	µg/kg		0.00			
6595	Pentachlorohexane	µg/kg		0.00			
6717	Tetrachlorobenzenes	µg/kg		0.00			
6705	1,2,3,4-Tetrachlorobenzene	µg/kg		0.00			
6710	1,2,3,5-Tetrachlorobenzene	µg/kg		0.00			
6715	1,2,4,5-Tetrachlorobenzene	µg/kg		0.00			
6730	2,3,4,5-Tetrachlorophenol	µg/kg		0.00			
6735	2,3,4,6-Tetrachlorophenol	µg/kg		0.00			
6740	2,3,5,6-Tetrachlorophenol	µg/kg		0.00			
5150	1,2,3-Trichlorobenzene	µg/kg		0.00			
6800	1,3,5-Trichlorobenzene	µg/kg		0.00			
6845	2,3,4-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6850	2,3,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6855	2,3,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6860	2,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-50 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/29/05
Study Dates: 04/25/05 - 06/09/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Additional Base / Neutrals and Acids in Soil (Cont.)							
6865	2,4,6-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
6870	3,4,5-Trichlorophenyl-4-nitrophenylether	µg/kg		0.00			
Pesticides in Soil							
7025	Aldrin	µg/kg	109	151	45.8 - 198	Acceptable	EPA 8081A
7110	alpha-BHC	µg/kg	343	466	117 - 641	Acceptable	EPA 8081A
7115	beta-BHC	µg/kg	287	486	169 - 613	Acceptable	EPA 8081A
7105	delta-BHC	µg/kg	139	204	70.9 - 273	Acceptable	EPA 8081A
7120	gamma-BHC(Lindane)	µg/kg	95.6	141	37.4 - 195	Acceptable	EPA 8081A
7240	alpha-Chlordane	µg/kg	60.4	82.2	30.0 - 108	Acceptable	EPA 8081A
7245	gamma-Chlordane	µg/kg	64.8	84.7	33.5 - 116	Acceptable	EPA 8081A
7355	4,4'-DDD	µg/kg	232	320	128 - 432	Acceptable	EPA 8081A
7360	4,4'-DDE	µg/kg	163	221	80.6 - 313	Acceptable	EPA 8081A
7365	4,4'-DDT	µg/kg	113	209	36.0 - 294	Acceptable	EPA 8081A
7470	Dieldrin	µg/kg	289	354	150 - 474	Acceptable	EPA 8081A
7540	Endrin	µg/kg	81.9	96.6	33.8 - 150	Acceptable	EPA 8081A
7530	Endrin aldehyde	µg/kg	151	294	35.3 - 389	Acceptable	EPA 8081A
7535	Endrin ketone	µg/kg	84.9	115	37.5 - 162	Acceptable	EPA 8081A
7510	Endosulfan I	µg/kg	89.5	166	27.5 - 187	Acceptable	EPA 8081A
7515	Endosulfan II	µg/kg	129	228	60.1 - 284	Acceptable	EPA 8081A
7520	Endosulfan sulfate	µg/kg	63.8	109	21.3 - 170	Acceptable	EPA 8081A
7685	Heptachlor	µg/kg	175	230	69.5 - 313	Acceptable	EPA 8081A
7690	Heptachlor epoxide	µg/kg	346	453	143 - 631	Acceptable	EPA 8081A
7810	Methoxychlor	µg/kg	95.0	132	1.70 - 218	Acceptable	EPA 8081A
Chlordane in Soil							
7250	Chlordane, Technical	µg/kg	178	262	55.3 - 331	Acceptable	EPA 8081A
Toxaphene in Soil							
8250	Toxaphene	µg/kg	325	358	0.00 - 498	Acceptable	EPA 8081A
Volatiles in Soil							
4315	Acetone	µg/kg	115	136	0.900 - 245	Acceptable	EPA 8260B
4320	Acetonitrile	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4325	Acrolein	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4375	Benzene	µg/kg	115	123	65.8 - 176	Acceptable	EPA 8260B
4395	Bromodichloromethane	µg/kg	149	159	112 - 223	Acceptable	EPA 8260B
4400	Bromoform	µg/kg	153	164	105 - 233	Acceptable	EPA 8260B

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)							
4950	Bromomethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4410	2-Butanone (MEK)	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4450	Carbon disulfide	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4455	Carbon tetrachloride	µg/kg	84.0	94.5	46.5 - 147	Acceptable	EPA 8260B
4475	Chlorobenzene	µg/kg	79.8	86.0	55.2 - 118	Acceptable	EPA 8260B
4575	Chlorodibromomethane	µg/kg	32.2	30.4	19.1 - 41.6	Acceptable	EPA 8260B
4485	Chloroethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4500	2-Chloroethylvinylether	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4505	Chloroform	µg/kg	93.8	113	69.2 - 163	Acceptable	EPA 8260B
4960	Chloromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4570	1,2-Dibromo-3-chloropropane (DBCP)	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4585	1,2-Dibromoethane (EDB)	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4595	Dibromomethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4610	1,2-Dichlorobenzene	µg/kg	118	128	80.3 - 176	Acceptable	EPA 8260B
4615	1,3-Dichlorobenzene	µg/kg	119	130	72.6 - 185	Acceptable	EPA 8260B
4620	1,4-Dichlorobenzene	µg/kg	64.0	69.0	42.5 - 99.8	Acceptable	EPA 8260B
4625	Dichlorodifluoromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4630	1,1-Dichloroethane	µg/kg	112	125	72.4 - 182	Acceptable	EPA 8260B
4635	1,2-Dichloroethane	µg/kg	54.3	60.9	35.1 - 86.3	Acceptable	EPA 8260B
4640	1,1-Dichloroethylene	µg/kg	79.5	82.0	39.9 - 140	Acceptable	EPA 8260B
4645	cis-1,2-Dichloroethylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4700	trans-1,2-Dichloroethylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4655	1,2-Dichloropropane	µg/kg	144	158	101 - 217	Acceptable	EPA 8260B
4680	cis-1,3-Dichloropropylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4685	trans-1,3-Dichloropropylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4765	Ethylbenzene	µg/kg	59.6	64.5	35.5 - 91.5	Acceptable	EPA 8260B
4860	2-Hexanone	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4975	Methylene chloride	µg/kg	123	143	66.6 - 209	Acceptable	EPA 8260B
4995	4-Methyl-2-pentanone (MIBK)	µg/kg	131	121	54.1 - 176	Acceptable	EPA 8260B
5100	Styrene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5105	1,1,1,2-Tetrachloroethane	µg/kg	172	164	105 - 229	Acceptable	EPA 8260B
5110	1,1,2,2-Tetrachloroethane	µg/kg	85.9	73.1	40.4 - 100	Acceptable	EPA 8260B
5115	Tetrachloroethylene	µg/kg	195	184	87.1 - 269	Acceptable	EPA 8260B
5140	Toluene	µg/kg	61.4	64.5	38.4 - 90.4	Acceptable	EPA 8260B
5160	1,1,1-Trichloroethane	µg/kg	122	134	69.7 - 202	Acceptable	EPA 8260B
5165	1,1,2-Trichloroethane	µg/kg	27.3	28.4	18.3 - 37.9	Acceptable	EPA 8260B
5170	Trichloroethylene	µg/kg	139	149	80.9 - 223	Acceptable	EPA 8260B

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

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SOIL-50 Final Complete Report

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Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/29/05
Study Dates: 04/25/05 - 06/09/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)							
5175	Trichlorofluoromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5180	1,2,3-Trichloropropane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5225	Vinyl acetate	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5235	Vinyl chloride	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5260	Total, Xylenes	µg/kg	300	324	156 - 494	Acceptable	EPA 8260B
Herbicides in Soil							
8545	2,4-D	µg/kg	8.68	390	0.00 - 648	Acceptable	EPA 8151A
8555	Dalapon	µg/kg	< 16.7	0.00		Acceptable	EPA 8151A
8560	2,4-DB	µg/kg	159	1270	0.00 - 2150	Acceptable	EPA 8151A
8595	Dicamba	µg/kg	11.6	381	0.00 - 591	Acceptable	EPA 8151A
8620	Dinoseb	µg/kg	547	2070	0.00 - 2340	Acceptable	EPA 8151A
7780	MCPP	µg/kg	322	0.00		Not Acceptable	EPA 8151A
6500	4-Nitrophenol	µg/kg	385	1250	0.00 - 1970	Acceptable	EPA 8151A
6605	Pentachlorophenol	µg/kg	276	931	0.00 - 1290	Acceptable	EPA 8151A
8655	2,4,5-T	µg/kg	10.2	208	0.00 - 351	Acceptable	EPA 8151A
8650	2,4,5-TP (Silvex)	µg/kg	26.4	431	0.00 - 684	Acceptable	EPA 8151A
PCBs in Soil							
8880	Aroclor 1016	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8885	Aroclor 1221	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8890	Aroclor 1232	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8895	Aroclor 1242	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8900	Aroclor 1248	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8905	Aroclor 1254	mg/kg	4.34	4.49	1.31 - 5.87	Acceptable	EPA 8082A
8910	Aroclor 1260	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A

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Report Issued: 06/29/05
Study Dates: 04/25/05 - 06/09/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
PAHs by HPLC in Soil							
5500	Acenaphthene	µg/kg	216	366	75.0 - 387	Acceptable	EPA 8310 UV
5505	Acenaphthylene	µg/kg	84.7	138	9.50 - 138	Acceptable	EPA 8310 UV
5555	Anthracene	µg/kg	85.6	195	28.4 - 195	Acceptable	EPA 8310 UV
5575	Benzo(a)anthracene	µg/kg	42.7	58.4	15.7 - 70.7	Acceptable	EPA 8310 UV
5585	Benzo(b)fluoranthene	µg/kg	111	145	40.2 - 170	Acceptable	EPA 8310 UV
5600	Benzo(k)fluoranthene	µg/kg	102	140	38.2 - 159	Acceptable	EPA 8310 UV
5590	Benzo(g,h,i)perylene	µg/kg	55.1	86.3	6.50 - 123	Acceptable	EPA 8310 UV
5580	Benzo(a)pyrene	µg/kg	36.3	54.0	13.3 - 60.9	Acceptable	EPA 8310 UV
5855	Chrysene	µg/kg	71.7	92.7	44.2 - 101	Acceptable	EPA 8310 UV
5895	Dibenz(a,h)anthracene	µg/kg	142	188	79.4 - 213	Acceptable	EPA 8310 UV
6265	Fluoranthene	µg/kg	64.0	73.5	29.9 - 94.1	Acceptable	EPA 8310 UV
6270	Fluorene	µg/kg	240	404	106 - 404	Acceptable	EPA 8310 UV
6315	Indeno(1,2,3-cd)pyrene	µg/kg	99.9	146	28.0 - 172	Acceptable	EPA 8310 UV
5005	Naphthalene	µg/kg	216	517	52.7 - 617	Acceptable	EPA 8310 UV
6615	Phenanthrene	µg/kg	68.5	94.5	33.6 - 101	Acceptable	EPA 8310 UV
6665	Pyrene	µg/kg	53.4	64.5	19.6 - 82.0	Acceptable	EPA 8310 UV
Nitroaromatics & Nitramines in Soil							
9306	4-Amino-2,6-dinitrotoluene	µg/kg	2720	3620	1160 - 3620	Acceptable	EPA 8330
9303	2-Amino-4,6-dinitrotoluene	µg/kg	2600	2910	1450 - 3470	Acceptable	EPA 8330
6160	1,3-Dinitrobenzene	µg/kg	9360	9110	4930 - 11800	Acceptable	EPA 8330
6185	2,4-Dinitrotoluene	µg/kg	4070	3890	2230 - 5210	Acceptable	EPA 8330
6190	2,6-Dinitrotoluene	µg/kg	6170	6120	3000 - 8100	Acceptable	EPA 8330
9522	HMX	µg/kg	4420	5000	2210 - 6710	Acceptable	EPA 8330
5015	Nitrobenzene	µg/kg	7960	7710	4440 - 9700	Acceptable	EPA 8330
9507	2-Nitrotoluene	µg/kg	7190	7110	4340 - 9320	Acceptable	EPA 8330
9510	3-Nitrotoluene	µg/kg	3260	2970	1860 - 4100	Acceptable	EPA 8330
9513	4-Nitrotoluene	µg/kg	5230	4950	2460 - 7200	Acceptable	EPA 8330
9432	RDX	µg/kg	7820	9500	4840 - 11600	Acceptable	EPA 8330
6415	Tetryl	µg/kg	< 300	0.00		Acceptable	EPA 8330
6885	1,3,5-Trinitrobenzene	µg/kg	8670	9050	3810 - 12000	Acceptable	EPA 8330
9651	2,4,6-Trinitrotoluene	µg/kg	8320	9600	4540 - 10700	Acceptable	EPA 8330
PAHs in Soil							
5500	Acenaphthene	µg/kg	2190	3520	685 - 3520	Acceptable	EPA 8310 UV
5505	Acenaphthylene	µg/kg	716	4610	0.00 - 4610	Acceptable	EPA 8310 UV
5555	Anthracene	µg/kg	556	1600	133 - 1600	Acceptable	EPA 8310 UV

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-50 Final Complete Report

Tenkasi S. Viswanathan
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EPA ID: KS00902
ERA Laboratory Code: A5230-01
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Study Dates: 04/25/05 - 06/09/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
PAHs in Soil (Cont.)							
5575	Benzo(a)anthracene	µg/kg	4710	6300	1760 - 7000	Acceptable	EPA 8310 UV
5585	Benzo(b)fluoranthene	µg/kg	1030	1180	398 - 1460	Acceptable	EPA 8310 UV
5600	Benzo(k)fluoranthene	µg/kg	4480	5500	1690 - 5950	Acceptable	EPA 8310 UV
5590	Benzo(g,h,i)perylene	µg/kg	462	686	65.0 - 845	Acceptable	EPA 8310 UV
5580	Benzo(a)pyrene	µg/kg	1760	7890	156 - 7890	Acceptable	EPA 8310 UV
5855	Chrysene	µg/kg	2370	3040	1130 - 3410	Acceptable	EPA 8310 UV
5895	Dibenz(a,h)anthracene	µg/kg	841	992	306 - 1180	Acceptable	EPA 8310 UV
6265	Fluoranthene	µg/kg	6320	7930	2160 - 9240	Acceptable	EPA 8310 UV
6270	Fluorene	µg/kg	4050	5500	1720 - 5860	Acceptable	EPA 8310 UV
6315	Indeno(1,2,3-cd)pyrene	µg/kg	476	593	157 - 735	Acceptable	EPA 8310 UV
5005	Naphthalene	µg/kg	467	888	0.00 - 1220	Acceptable	EPA 8310 UV
6615	Phenanthrene	µg/kg	2640	3310	1230 - 3610	Acceptable	EPA 8310 UV
6665	Pyrene	µg/kg	4640	6200	1770 - 6550	Acceptable	EPA 8310 UV
Ignitability							
1780	Ignitability (Flashpoint)	deg. F	132	135	116 - 146	Acceptable	EPA 1010
Corrosivity (pH)							
1625	Corrosivity (pH)	S.U.	10.1	9.76	9.08 - 10.1	Acceptable	EPA 9045C

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

ENVIRONMENTAL RESOURCE ASSOCIATES®

Study: **SOIL-50**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method B**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil							
1000	Aluminum	mg/kg	3840	8010	2820 - 11500	Acceptable	EPA 6020
1005	Antimony	mg/kg	143	171	0.00 - 171	Acceptable	EPA 6020
1010	Arsenic	mg/kg	176	187	129 - 223	Acceptable	EPA 6020
1015	Barium	mg/kg	336	372	275 - 443	Acceptable	EPA 6020
1020	Beryllium	mg/kg	111	126	90.2 - 148	Acceptable	EPA 6020
1025	Boron	mg/kg	99.1	141	67.4 - 159	Acceptable	EPA 6020
1030	Cadmium	mg/kg	59.2	61.5	41.8 - 69.2	Acceptable	EPA 6020
1035	Calcium	mg/kg	3770	4190	2850 - 4990	Acceptable	EPA 6020
1040	Chromium	mg/kg	109	122	82.7 - 143	Acceptable	EPA 6020
1050	Cobalt	mg/kg	137	150	94.2 - 164	Acceptable	EPA 6020
1055	Copper	mg/kg	78.0	85.6	60.3 - 99.6	Acceptable	EPA 6020
1070	Iron	mg/kg	11800	15300	2820 - 24200	Acceptable	EPA 6020
1075	Lead	mg/kg	131	133	93.5 - 157	Acceptable	EPA 6020
1085	Magnesium	mg/kg	2330	2840	1860 - 3510	Acceptable	EPA 6020
1090	Manganese	mg/kg	267	267	188 - 332	Acceptable	EPA 6020
1095	Mercury	mg/kg	2.24	2.39	1.43 - 3.53	Acceptable	EPA 6020
1100	Molybdenum	mg/kg	134	141	90.7 - 157	Acceptable	EPA 6020
1105	Nickel	mg/kg	162	186	124 - 206	Acceptable	EPA 6020
1125	Potassium	mg/kg	2450	2820	1800 - 4050	Acceptable	EPA 6020
1140	Selenium	mg/kg	124	127	83.6 - 148	Acceptable	EPA 6020
1150	Silver	mg/kg	69.5	70.7	47.0 - 84.8	Acceptable	EPA 6020
1155	Sodium	mg/kg	481	509	323 - 631	Acceptable	EPA 6020
1160	Strontium	mg/kg	106	113	75.4 - 137	Acceptable	EPA 6020
1165	Thallium	mg/kg	107	113	71.1 - 133	Acceptable	EPA 6020
1175	Tin	mg/kg	156	153	89.1 - 187	Acceptable	EPA 6020
1180	Titanium	mg/kg	167	320	0.00 - 601	Acceptable	EPA 6020
1185	Vanadium	mg/kg	84.7	91.0	48.2 - 110	Acceptable	EPA 6020
1190	Zinc	mg/kg	164	181	122 - 214	Acceptable	EPA 6020

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
PAHs by HPLC in Soil							
5500	Acenaphthene	µg/kg	243	366	75.0 - 387	Acceptable	EPA 8270 SIM
5505	Acenaphthylene	µg/kg	62.8	138	9.50 - 138	Acceptable	EPA 8270 SIM
5555	Anthracene	µg/kg	86.7	195	28.4 - 195	Acceptable	EPA 8270 SIM
5575	Benzo(a)anthracene	µg/kg	51.4	58.4	15.7 - 70.7	Acceptable	EPA 8270 SIM
5585	Benzo(b)fluoranthene	µg/kg	130	145	40.2 - 170	Acceptable	EPA 8270 SIM
5600	Benzo(k)fluoranthene	µg/kg	96.3	140	38.2 - 159	Acceptable	EPA 8270 SIM
5590	Benzo(g,h,i)perylene	µg/kg	66.1	86.3	6.50 - 123	Acceptable	EPA 8270 SIM
5580	Benzo(a)pyrene	µg/kg	42.0	54.0	13.3 - 60.9	Acceptable	EPA 8270 SIM
5855	Chrysene	µg/kg	73.1	92.7	44.2 - 101	Acceptable	EPA 8270 SIM
5895	Dibenz(a,h)anthracene	µg/kg	158	188	79.4 - 213	Acceptable	EPA 8270 SIM
6265	Fluoranthene	µg/kg	72.4	73.5	29.9 - 94.1	Acceptable	EPA 8270 SIM
6270	Fluorene	µg/kg	299	404	106 - 404	Acceptable	EPA 8270 SIM
6315	Indeno(1,2,3-cd)pyrene	µg/kg	136	146	28.0 - 172	Acceptable	EPA 8270 SIM
5005	Naphthalene	µg/kg	388	517	52.7 - 617	Acceptable	EPA 8270 SIM
6615	Phenanthrene	µg/kg	71.7	94.5	33.6 - 101	Acceptable	EPA 8270 SIM
6665	Pyrene	µg/kg	60.2	64.5	19.6 - 82.0	Acceptable	EPA 8270 SIM
PAHs in Soil							
5500	Acenaphthene	µg/kg	2230	3520	685 - 3520	Acceptable	EPA 8270 SIM
5505	Acenaphthylene	µg/kg	591	4610	0.00 - 4610	Acceptable	EPA 8270 SIM
5555	Anthracene	µg/kg	540	1600	133 - 1600	Acceptable	EPA 8270 SIM
5575	Benzo(a)anthracene	µg/kg	4290	6300	1760 - 7000	Acceptable	EPA 8270 SIM
5585	Benzo(b)fluoranthene	µg/kg	996	1180	398 - 1460	Acceptable	EPA 8270 SIM
5600	Benzo(k)fluoranthene	µg/kg	3560	5500	1690 - 5950	Acceptable	EPA 8270 SIM
5590	Benzo(g,h,i)perylene	µg/kg	499	686	65.0 - 845	Acceptable	EPA 8270 SIM
5580	Benzo(a)pyrene	µg/kg	1760	7890	156 - 7890	Acceptable	EPA 8270 SIM
5855	Chrysene	µg/kg	2110	3040	1130 - 3410	Acceptable	EPA 8270 SIM
5895	Dibenz(a,h)anthracene	µg/kg	900	992	306 - 1180	Acceptable	EPA 8270 SIM
6265	Fluoranthene	µg/kg	6110	7930	2160 - 9240	Acceptable	EPA 8270 SIM
6270	Fluorene	µg/kg	4190	5500	1720 - 5860	Acceptable	EPA 8270 SIM
6315	Indeno(1,2,3-cd)pyrene	µg/kg	640	593	157 - 735	Acceptable	EPA 8270 SIM
5005	Naphthalene	µg/kg	656	888	0.00 - 1220	Acceptable	EPA 8270 SIM
6615	Phenanthrene	µg/kg	2490	3310	1230 - 3610	Acceptable	EPA 8270 SIM
6665	Pyrene	µg/kg	4800	6200	1770 - 6550	Acceptable	EPA 8270 SIM

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

ENVIRONMENTAL RESOURCE ASSOCIATES® SOIL-52 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 **EPA ID: KS00902**

Report Issued: 12/29/05
Study Dates: 10/24/05 - 12/08/05

SOIL Study Definitions:

The reported value is the value that the laboratory reported to ERA.

The ERA assigned value for the Organic Proficiency Testing Standards is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation as applicable. The ERA assigned value for the Inorganic Proficiency Testing Standards, with the exception of the TCLP Metals in Soil, is equal to the maximum amount of the parameter available in the standard by applicable EPA methodologies. The ERA assigned value for the TCLP metals is equal to the mean of ERA's internal initial analytical analyses. All NELAC parameters not added to a standard are given an assigned Value of "0", per the guidance issued by the NELAC Board of Directors, on December 14, 2000. Non-NELAC parameters not added to a standard may be given an assigned value of less than a minimum verified concentration as determined in the background soil for applicable EPA methodologies.

The acceptance limits are established per the NELAC PT program criteria or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable.

The Performance Evaluation:

Acceptable = Reported Value falls within the Acceptance Limits.

Not Acceptable = Reported Value falls outside of the Acceptance Limits.

No Evaluation = Reported Value cannot be evaluated.

The method description is the method the laboratory reported to ERA.

Any Performance Evaluation left blank indicates results were evaluated as 'Not Reported'.

SOIL Study Discussion:

ERA's Proficiency Testing (PT) Study, SOIL-52, has been reviewed by ERA Senior Management and certified compliant with the requirements of the National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program Standards, Chapter 2, July 2003.

Per the requirements of the NELAC Proficiency Testing Program, a full review of all accuracy verification, homogeneity and stability data was completed. All analytical verification data for all analytes in the SOIL-52 standards met the acceptance criteria contained in the NELAC Proficiency Testing Program Standards, Chapter 2, July 2003.

During the review of the SOIL-52 data ERA observed multiple analytes whose NELAC calculated upper acceptance limits were determined to be a value less than the assigned value for the analyte. Per the NELAC Standards, Chapter 2, Appendix C, and the NELAC PT Fields of Testing - RCRA - Solid, the acceptance criteria for soil studies are to be set to ± 3 standard deviations about the mean of the study data. The NELAC Standards, Chapter 2, do not address the situation when the upper acceptance limit is determined to be less than the assigned value. To minimize the possibility of penalizing those laboratories who routinely use methods designed to improve recoveries of these analytes, ERA has set the upper acceptance limits to the assigned value. ERA has communicated this to the NELAC PT Board.

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's statistical review of the data there was one anomaly noted. This anomaly is addressed on the following page

The Proficiency Testing Study, SOIL-52, report shall not be reproduced except in its entirety and not without the permission of the participating laboratory. The report must not be used by the participating laboratory to claim product endorsement by NVLAP or any agency

NVLAP® Lab code: 200355-0

SOIL Study Definitions:

SOIL Study Discussion:

of the U. S. government.

If you have any questions regarding ERA's Proficiency Testing Study, SOIL-52, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP®
Lab code: 200086-0

ERA Laboratory Code: A5230-01 **EPA ID: KS00902**

Report Issued: 12/29/05
Study Dates: 10/24/05 - 12/08/05

Study Discussion: Soil-52 Metals in Soil - Silver

During ERA's review of the results reported for the Metals in Soil standard in the Soil-52 study, we observed a failure rate of 18.2% for silver, with almost all of the 'Not Acceptable' results being reported low. The high failure rate related to low recoveries caused us to initiate a careful review of both our manufacturing and internal analytical verification data for accuracy, homogeneity and stability. The review of all internal data showed that the assigned value and acceptance limits for silver are accurate and that this analyte was stable during the course of the study.

In further reviewing the study data, it was found that 32.4% of the low-end not acceptable values for silver were associated with HNO3-only digestion techniques. As mentioned in USEPA methods 3050B and 3051A, a digestion utilizing hydrochloric acid in addition to nitric acid is generally required to achieve acceptable recoveries of silver, as well as several other metals. ERA internal research as well as historical study data review confirms that HNO3-only digestions provide inadequate recoveries for silver.

If you would like to discuss this situation further, please feel free to call ERA's Inorganic Chemistry department at 1-800-372-0122.

NVLAP® Lab code: 200086-0

ENVIRONMENTAL
RESOURCE ASSOCIATES®

Study: **SOIL-52**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method A**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-52 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/29/05
Study Dates: 10/24/05 - 12/08/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil							
1000	Aluminum	mg/kg	4000	8160	2390 - 11900	Acceptable	EPA 200.7
1005	Antimony	mg/kg	176	213	0.00 - 233	Acceptable	EPA 200.7
1010	Arsenic	mg/kg	197	226	143 - 254	Acceptable	EPA 200.7
1015	Barium	mg/kg	621	680	469 - 809	Acceptable	EPA 200.7
1020	Beryllium	mg/kg	73.2	75.8	48.4 - 90.2	Acceptable	EPA 200.7
1025	Boron	mg/kg	93.3	127	60.5 - 139	Acceptable	EPA 200.7
1030	Cadmium	mg/kg	87.6	88.1	53.7 - 103	Acceptable	EPA 200.7
1035	Calcium	mg/kg	3910	4380	2790 - 5370	Acceptable	EPA 200.7
1040	Chromium	mg/kg	133	142	90.9 - 167	Acceptable	EPA 200.7
1050	Cobalt	mg/kg	124	135	81.5 - 151	Acceptable	EPA 200.7
1055	Copper	mg/kg	105	102	66.9 - 121	Acceptable	EPA 200.7
1070	Iron	mg/kg	10600	15300	620 - 27400	Acceptable	EPA 200.7
1075	Lead	mg/kg	105	113	75.7 - 134	Acceptable	EPA 200.7
1085	Magnesium	mg/kg	2040	2590	1560 - 3220	Acceptable	EPA 200.7
1090	Manganese	mg/kg	328	337	230 - 420	Acceptable	EPA 200.7
1095	Mercury	mg/kg	4.58	3.86	2.28 - 5.52	Acceptable	EPA 7471A
1100	Molybdenum	mg/kg	177	188	117 - 211	Acceptable	EPA 200.7
1105	Nickel	mg/kg	223	237	149 - 273	Acceptable	EPA 200.7
1125	Potassium	mg/kg	1960	2450	1400 - 3560	Acceptable	EPA 200.7
1140	Selenium	mg/kg	109	116	68.6 - 139	Acceptable	EPA 200.7
1150	Silver	mg/kg	137	172	84.6 - 221	Acceptable	EPA 200.7
1155	Sodium	mg/kg	760	776	473 - 981	Acceptable	EPA 200.7
1160	Strontium	mg/kg	72.2	69.1	44.0 - 92.6	Acceptable	EPA 200.7
1165	Thallium	mg/kg	144	149	90.7 - 175	Acceptable	EPA 200.7
1175	Tin	mg/kg	124	138	77.8 - 170	Acceptable	EPA 200.7
1180	Titanium	mg/kg	181	320	0.00 - 547	Acceptable	EPA 200.7
1185	Vanadium	mg/kg	145	165	95.6 - 196	Acceptable	EPA 200.7
1190	Zinc	mg/kg	155	164	101 - 199	Acceptable	EPA 200.7
Cyanide in Soil							
1635	Cyanide, total	mg/kg	161	284	0.00 - 432	Acceptable	EPA 9010B
1640	Reactive Cyanide	mg/kg	< 12	< 25.0	< 25.0	Acceptable	EPA 9010B
Hexavalent Chromium in Soil							
1045	Chromium VI	mg/kg	56.4	143	16.3 - 143	Acceptable	EPA 7196A

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

ENVIRONMENTAL RESOURCE ASSOCIATES

SOIL-52 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
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Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/29/05
Study Dates: 10/24/05 - 12/08/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil							
5500	Acenaphthene	µg/kg	3330	4330	1160 - 5260	Acceptable	EPA 8270C
5505	Acenaphthylene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5145	2-Amino-1-methylbenzene (o-toluidine)	µg/kg		0.00			
5545	Aniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5555	Anthracene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5595	Benzidine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5610	Benzoic acid	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5575	Benzo(a)anthracene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5585	Benzo(b)fluoranthene	µg/kg	2910	3650	1200 - 4520	Acceptable	EPA 8270C
5600	Benzo(k)fluoranthene	µg/kg	2190	2800	1100 - 3340	Acceptable	EPA 8270C
5590	Benzo(g,h,i)perylene	µg/kg	3780	4390	1480 - 5720	Acceptable	EPA 8270C
5580	Benzo(a)pyrene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5630	Benzyl alcohol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5660	4-Bromophenyl-phenylether	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5670	Butylbenzylphthalate	µg/kg	2990	3460	1140 - 4680	Acceptable	EPA 8270C
5680	Carbazole	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5745	4-Chloroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5760	bis(2-Chloroethoxy)methane	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5765	bis(2-Chloroethyl)ether	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5780	bis(2-Chloroisopropyl)ether	µg/kg	1300	4360	596 - 5260	Acceptable	EPA 8270C
5700	4-Chloro-3-methylphenol	µg/kg	3710	6900	1090 - 7210	Acceptable	EPA 8270C
5790	1-Chloronaphthalene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5795	2-Chloronaphthalene	µg/kg	1470	1820	448 - 2210	Acceptable	EPA 8270C
5800	2-Chlorophenol	µg/kg	2360	4330	654 - 4330	Acceptable	EPA 8270C
5825	4-Chlorophenyl-phenylether	µg/kg	2920	3610	976 - 4440	Acceptable	EPA 8270C
5855	Chrysene	µg/kg	3040	3510	1450 - 4790	Acceptable	EPA 8270C
5895	Dibenz(a,h)anthracene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5905	Dibenzofuran	µg/kg	4850	6300	1490 - 7670	Acceptable	EPA 8270C
5925	Di-n-butylphthalate	µg/kg	4040	4160	1450 - 5250	Acceptable	EPA 8270C
4610	1,2-Dichlorobenzene	µg/kg	4910	8610	0.00 - 9720	Acceptable	EPA 8270C
4615	1,3-Dichlorobenzene	µg/kg	2580	4380	0.00 - 4960	Acceptable	EPA 8270C
4620	1,4-Dichlorobenzene	µg/kg	3850	6640	0.00 - 7270	Acceptable	EPA 8270C
5945	3,3'-Dichlorobenzidine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6000	2,4-Dichlorophenol	µg/kg	1520	2790	376 - 3000	Acceptable	EPA 8270C
6005	2,6-Dichlorophenol	µg/kg	2050	4440	456 - 4440	Acceptable	EPA 8270C

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-52 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/29/05
Study Dates: 10/24/05 - 12/08/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)							
6070	Diethylphthalate	µg/kg	1980	2390	729 - 2930	Acceptable	EPA 8270C
6130	2,4-Dimethylphenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6135	Dimethylphthalate	µg/kg	3290	4140	1190 - 4970	Acceptable	EPA 8270C
6360	4,6-Dinitro-2-methylphenol	µg/kg	680	6650	0.00 - 6650	Acceptable	EPA 8270C
6175	2,4-Dinitrophenol	µg/kg	< 125	5090	0.00 - 5090	Acceptable	EPA 8270C
6185	2,4-Dinitrotoluene	µg/kg	4060	6120	1550 - 7610	Acceptable	EPA 8270C
6190	2,6-Dinitrotoluene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6200	Di-n-octylphthalate	µg/kg	4610	5230	1500 - 7110	Acceptable	EPA 8270C
6255	bis(2-Ethylhexyl)phthalate	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6265	Fluoranthene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6270	Fluorene	µg/kg	4690	6100	1740 - 7360	Acceptable	EPA 8270C
6275	Hexachlorobenzene	µg/kg	5890	7310	2270 - 8990	Acceptable	EPA 8270C
4835	Hexachlorobutadiene	µg/kg	4030	5580	430 - 6610	Acceptable	EPA 8270C
6285	Hexachlorocyclopentadiene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
4840	Hexachloroethane	µg/kg	4070	6970	0.00 - 7640	Acceptable	EPA 8270C
6315	Indeno(1,2,3-cd)pyrene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6320	Isophorone	µg/kg	3850	5320	1180 - 6300	Acceptable	EPA 8270C
6385	2-Methylnaphthalene	µg/kg	2780	3790	710 - 4670	Acceptable	EPA 8270C
6400	2-Methylphenol	µg/kg	< 125	4900	0.00 - 4900	Acceptable	EPA 8270C
6410	4-Methylphenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5005	Naphthalene	µg/kg	3480	5200	601 - 5960	Acceptable	EPA 8270C
6460	2-Nitroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6465	3-Nitroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6470	4-Nitroaniline	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5015	Nitrobenzene	µg/kg	2840	4050	707 - 4670	Acceptable	EPA 8270C
6490	2-Nitrophenol	µg/kg	3750	7820	1060 - 8620	Acceptable	EPA 8270C
6500	4-Nitrophenol	µg/kg	3210	7470	180 - 8400	Acceptable	EPA 8270C
6525	N-Nitrosodiethylamine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6530	N-Nitrosodimethylamine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6535	N-Nitrosodiphenylamine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6545	N-Nitroso-di-n-propylamine	µg/kg	2630	3760	750 - 4590	Acceptable	EPA 8270C
6590	Pentachlorobenzene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6605	Pentachlorophenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6615	Phenanthrene	µg/kg	2900	3560	1390 - 4150	Acceptable	EPA 8270C
6625	Phenol	µg/kg	4350	7980	1090 - 8290	Acceptable	EPA 8270C

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-52 Final Complete Report

Tenkasi S. Viswanathan
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15130 S Keeler
Suite B
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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/29/05
Study Dates: 10/24/05 - 12/08/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Base / Neutrals and Acids in Soil (Cont.)							
6665	Pyrene	µg/kg	4590	5850	1350 - 7590	Acceptable	EPA 8270C
5095	Pyridine	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6715	1,2,4,5-Tetrachlorobenzene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6735	2,3,4,6-Tetrachlorophenol	µg/kg	< 125	0.00		Acceptable	EPA 8270C
5155	1,2,4-Trichlorobenzene	µg/kg	< 125	0.00		Acceptable	EPA 8270C
6835	2,4,5-Trichlorophenol	µg/kg	3100	4920	883 - 5380	Acceptable	EPA 8270C
6840	2,4,6-Trichlorophenol	µg/kg	4340	7210	1050 - 7410	Acceptable	EPA 8270C
Pesticides in Soil							
7025	Aldrin	µg/kg	243	356	20.5 - 486	Acceptable	EPA 8081A
7110	alpha-BHC	µg/kg	113	205	8.40 - 256	Acceptable	EPA 8081A
7115	beta-BHC	µg/kg	153	291	20.7 - 363	Acceptable	EPA 8081A
7105	delta-BHC	µg/kg	106	140	2.30 - 188	Acceptable	EPA 8081A
7120	gamma-BHC(Lindane)	µg/kg	84.5	133	3.70 - 166	Acceptable	EPA 8081A
7240	alpha-Chlordane	µg/kg	101	155	16.4 - 198	Acceptable	EPA 8081A
7245	gamma-Chlordane	µg/kg	71.0	115	14.2 - 144	Acceptable	EPA 8081A
7355	4,4'-DDD	µg/kg	225	352	34.9 - 483	Acceptable	EPA 8081A
7360	4,4'-DDE	µg/kg	172	246	46.9 - 315	Acceptable	EPA 8081A
7365	4,4'-DDT	µg/kg	94.6	150	15.5 - 211	Acceptable	EPA 8081A
7470	Dieldrin	µg/kg	150	318	35.5 - 417	Acceptable	EPA 8081A
7540	Endrin	µg/kg	174	205	17.9 - 280	Acceptable	EPA 8081A
7530	Endrin aldehyde	µg/kg	146	432	4.50 - 440	Acceptable	EPA 8081A
7535	Endrin ketone	µg/kg	262	392	24.5 - 508	Acceptable	EPA 8081A
7510	Endosulfan I	µg/kg	159	381	0.00 - 381	Acceptable	EPA 8081A
7515	Endosulfan II	µg/kg	113	265	0.00 - 265	Acceptable	EPA 8081A
7520	Endosulfan sulfate	µg/kg	192	314	22.4 - 420	Acceptable	EPA 8081A
7685	Heptachlor	µg/kg	132	209	0.600 - 275	Acceptable	EPA 8081A
7690	Heptachlor epoxide	µg/kg	165	250	28.2 - 314	Acceptable	EPA 8081A
7810	Methoxychlor	µg/kg	179	204	2.10 - 328	Acceptable	EPA 8081A
Chlordane in Soil							
7250	Chlordane, Technical	µg/kg	201	230	23.1 - 319	Acceptable	EPA 8081A
Toxaphene in Soil							
8250	Toxaphene	µg/kg	294	310	0.00 - 476	Acceptable	EPA 8081A

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-52 Final Complete Report

Tenkasi S. Viswanathan
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Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/29/05
Study Dates: 10/24/05 - 12/08/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil							
4315	Acetone	µg/kg	85.2	148	0.00 - 288	Acceptable	EPA 8260B
4320	Acetonitrile	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4325	Acrolein	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4375	Benzene	µg/kg	111	107	61.2 - 155	Acceptable	EPA 8260B
4395	Bromodichloromethane	µg/kg	93.4	95.8	57.1 - 139	Acceptable	EPA 8260B
4400	Bromoform	µg/kg	173	148	75.5 - 223	Acceptable	EPA 8260B
4950	Bromomethane	µg/kg	< 20	0.00		Acceptable	EPA 8260B
4410	2-Butanone (MEK)	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5000	tert-Butyl methyl ether (MTBE)	µg/kg	65.1	71.6	44.2 - 105	Acceptable	EPA 8260B
4450	Carbon disulfide	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4455	Carbon tetrachloride	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4475	Chlorobenzene	µg/kg	76.2	73.5	42.4 - 104	Acceptable	EPA 8260B
4575	Chlorodibromomethane	µg/kg	132	119	67.4 - 171	Acceptable	EPA 8260B
4485	Chloroethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4500	2-Chloroethylvinylether	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4505	Chloroform	µg/kg	33.2	32.4	17.0 - 49.0	Acceptable	EPA 8260B
4960	Chloromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4570	1,2-Dibromo-3-chloropropane (DBCP)	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4585	1,2-Dibromoethane (EDB)	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4595	Dibromomethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4610	1,2-Dichlorobenzene	µg/kg	174	156	88.7 - 215	Acceptable	EPA 8260B
4615	1,3-Dichlorobenzene	µg/kg	75.6	67.3	38.3 - 95.3	Acceptable	EPA 8260B
4620	1,4-Dichlorobenzene	µg/kg	158	149	82.2 - 206	Acceptable	EPA 8260B
4625	Dichlorodifluoromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4630	1,1-Dichloroethane	µg/kg	38.8	38.1	18.3 - 59.3	Acceptable	EPA 8260B
4635	1,2-Dichloroethane	µg/kg	98.8	93.7	51.4 - 137	Acceptable	EPA 8260B
4640	1,1-Dichloroethylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4645	cis-1,2-Dichloroethylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4700	trans-1,2-Dichloroethylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4655	1,2-Dichloropropane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4680	cis-1,3-Dichloropropylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4685	trans-1,3-Dichloropropylene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4765	Ethylbenzene	µg/kg	157	180	110 - 254	Acceptable	EPA 8260B
4860	2-Hexanone	µg/kg	< 10	0.00		Acceptable	EPA 8260B
4975	Methylene chloride	µg/kg	154	164	79.6 - 240	Acceptable	EPA 8260B

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-52 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/29/05
Study Dates: 10/24/05 - 12/08/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Volatiles in Soil (Cont.)							
4995	4-Methyl-2-pentanone (MIBK)	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5100	Styrene	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5105	1,1,1,2-Tetrachloroethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5110	1,1,2,2-Tetrachloroethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5115	Tetrachloroethylene	µg/kg	170	160	72.9 - 233	Acceptable	EPA 8260B
5140	Toluene	µg/kg	176	177	99.1 - 245	Acceptable	EPA 8260B
5160	1,1,1-Trichloroethane	µg/kg	28.0	30.1	14.2 - 47.4	Acceptable	EPA 8260B
5165	1,1,2-Trichloroethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5170	Trichloroethylene	µg/kg	156	151	81.3 - 219	Acceptable	EPA 8260B
5175	Trichlorofluoromethane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5180	1,2,3-Trichloropropane	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5225	Vinyl acetate	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5235	Vinyl chloride	µg/kg	< 10	0.00		Acceptable	EPA 8260B
5260	Total, Xylenes	µg/kg	154	196	93.7 - 304	Acceptable	EPA 8260B
Herbicides in Soil							
8505	Acifluorfen	µg/kg	610	1530	0.00 - 1530	Acceptable	EPA 8151A
8530	Bentazon	µg/kg	171	280	0.00 - 420	Acceptable	EPA 8151A
8540	Chloramben	µg/kg	< 3.3	285	0.00 - 467	Acceptable	EPA 8151A
8545	2,4-D	µg/kg	185	368	0.00 - 579	Acceptable	EPA 8151A
8560	2,4-DB	µg/kg	372	1050	0.00 - 1380	Acceptable	EPA 8151A
8550	Dacthal diacid (DCPA)	µg/kg		0.00			
8555	Dalapon	µg/kg	< 3.3	0.00		Acceptable	EPA 8151A
8595	Dicamba	µg/kg	185	340	0.00 - 520	Acceptable	EPA 8151A
8600	3,5-Dichlorobenzoic acid	µg/kg	< 3.3	0.00		Acceptable	EPA 8151A
8605	Dichlorprop	µg/kg	< 3.3	0.00		Acceptable	EPA 8151A
8620	Dinoseb	µg/kg	< 3.3	0.00		Acceptable	EPA 8151A
7775	MCPA	µg/kg	< 3.3	0.00		Acceptable	EPA 8151A
7780	MCPP	µg/kg	2010	0.00		Not Acceptable	EPA 8151A
6500	4-Nitrophenol	µg/kg	268	1040	0.00 - 1420	Acceptable	EPA 8151A
6605	Pentachlorophenol	µg/kg	493	931	0.00 - 1190	Acceptable	EPA 8151A
8645	Picloram	µg/kg	167	798	0.00 - 1080	Acceptable	EPA 8151A
8655	2,4,5-T	µg/kg	252	496	0.00 - 717	Acceptable	EPA 8151A
8650	2,4,5-TP (Silvex)	µg/kg	183	290	0.00 - 441	Acceptable	EPA 8151A

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
PCBs in Soil							
8880	Aroclor 1016	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8885	Aroclor 1221	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8890	Aroclor 1232	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8895	Aroclor 1242	mg/kg	14.4	15.2	0.00 - 23.7	Acceptable	EPA 8082A
8900	Aroclor 1248	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8905	Aroclor 1254	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A
8910	Aroclor 1260	mg/kg	< 0.05	0.00		Acceptable	EPA 8082A

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
PAHs by HPLC in Soil							
5500	Acenaphthene	µg/kg	103	152	29.5 - 170	Acceptable	EPA 8270 SIM
5505	Acenaphthylene	µg/kg	80.2	806	0.00 - 806	Acceptable	EPA 8270 SIM
5555	Anthracene	µg/kg	< 10	153	0.00 - 153	Acceptable	EPA 8270 SIM
5575	Benzo(a)anthracene	µg/kg	48.4	79.7	22.7 - 79.7	Acceptable	EPA 8270 SIM
5585	Benzo(b)fluoranthene	µg/kg	78.0	87.9	32.2 - 108	Acceptable	EPA 8270 SIM
5600	Benzo(k)fluoranthene	µg/kg	62.1	87.4	26.5 - 100	Acceptable	EPA 8270 SIM
5590	Benzo(g,h,i)perylene	µg/kg	65.5	84.3	19.8 - 101	Acceptable	EPA 8270 SIM
5580	Benzo(a)pyrene	µg/kg	24.6	156	9.21 - 156	Acceptable	EPA 8270 SIM
5855	Chrysene	µg/kg	39.0	57.9	20.8 - 70.0	Acceptable	EPA 8270 SIM
5895	Dibenz(a,h)anthracene	µg/kg	78.9	86.8	26.9 - 101	Acceptable	EPA 8270 SIM
6265	Fluoranthene	µg/kg	80.9	91.2	31.6 - 117	Acceptable	EPA 8270 SIM
6270	Fluorene	µg/kg	141	196	60.1 - 206	Acceptable	EPA 8270 SIM
6315	Indeno(1,2,3-cd)pyrene	µg/kg	77.7	69.2	22.5 - 82.3	Acceptable	EPA 8270 SIM
5005	Naphthalene	µg/kg	169	210	0.00 - 315	Acceptable	EPA 8270 SIM
6615	Phenanthrene	µg/kg	48.6	62.1	21.7 - 74.9	Acceptable	EPA 8270 SIM
6665	Pyrene	µg/kg	51.7	76.3	26.0 - 81.4	Acceptable	EPA 8270 SIM
Nitroaromatics & Nitramines in Soil							
9306	4-Amino-2,6-dinitrotoluene	µg/kg	3650	5040	1360 - 7020	Acceptable	EPA 8330
9303	2-Amino-4,6-dinitrotoluene	µg/kg	< 125	0.00		Acceptable	EPA 8330
6160	1,3-Dinitrobenzene	µg/kg	4360	4870	2570 - 6750	Acceptable	EPA 8330
6185	2,4-Dinitrotoluene	µg/kg	3040	3340	1640 - 4740	Acceptable	EPA 8330
6190	2,6-Dinitrotoluene	µg/kg	4710	5630	1420 - 9460	Acceptable	EPA 8330
9522	HMX	µg/kg	4400	6670	1920 - 9960	Acceptable	EPA 8330
5015	Nitrobenzene	µg/kg	4370	5100	2410 - 6470	Acceptable	EPA 8330
9507	2-Nitrotoluene	µg/kg	7120	8270	3460 - 11700	Acceptable	EPA 8330
9510	3-Nitrotoluene	µg/kg	2680	2970	798 - 5140	Acceptable	EPA 8330
9513	4-Nitrotoluene	µg/kg	4210	4950	1710 - 7610	Acceptable	EPA 8330
9432	RDX	µg/kg	16900	25700	8570 - 33200	Acceptable	EPA 8330
6415	Tetryl	µg/kg	< 125	0.00		Acceptable	EPA 8330
6885	1,3,5-Trinitrobenzene	µg/kg	10900	13300	3230 - 18800	Acceptable	EPA 8330
9651	2,4,6-Trinitrotoluene	µg/kg	2540	3330	1300 - 3980	Acceptable	EPA 8330
Ignitability							
1780	Ignitability (Flashpoint)	deg. F	102	120	108 - 140	Not Acceptable	EPA 1010

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-52 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/29/05
Study Dates: 10/24/05 - 12/08/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Corrosivity (pH)							
1625	Corrosivity (pH)	S.U.	7.96	7.15	6.53 - 7.81	Not Acceptable	EPA 9045C
TCLP Metals in Soil							
1005	Antimony	mg/L	1.62	1.64	0.930 - 2.07	Acceptable	EPA 6010B
1010	Arsenic	mg/L	1.47	1.51	0.953 - 2.03	Acceptable	EPA 6010B
1015	Barium	mg/L	3.03	3.59	2.24 - 3.88	Acceptable	EPA 6010B
1020	Beryllium	mg/L	2.19	2.57	1.47 - 3.21	Acceptable	EPA 6010B
1030	Cadmium	mg/L	1.70	1.83	1.26 - 2.14	Acceptable	EPA 6010B
1040	Chromium	mg/L	2.52	2.81	1.71 - 3.11	Acceptable	EPA 6010B
1075	Lead	mg/L	2.38	2.83	1.45 - 3.53	Acceptable	EPA 6010B
1095	Mercury	mg/L	0.752	0.523	0.428 - 1.02	Acceptable	EPA 7471A
1105	Nickel	mg/L	4.54	4.91	3.37 - 5.47	Acceptable	EPA 6010B
1140	Selenium	mg/L	2.93	3.04	1.76 - 3.71	Acceptable	EPA 6010B
1150	Silver	mg/L	0.435	0.350	0.0380 - 0.650	Acceptable	EPA 6010B
1190	Zinc	mg/L	2.55	3.29	1.49 - 3.49	Acceptable	EPA 6010B

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

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Study: **SOIL-52**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method B**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

SOIL-52 Final Complete Report

Tenkasi S. Viswanathan
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Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/29/05
Study Dates: 10/24/05 - 12/08/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Trace Metals in Soil							
1000	Aluminum	mg/kg	4110	8160	2390 - 11900	Acceptable	EPA 6020
1005	Antimony	mg/kg	174	213	0.00 - 233	Acceptable	EPA 6020
1010	Arsenic	mg/kg	210	226	143 - 254	Acceptable	EPA 6020
1015	Barium	mg/kg	637	680	469 - 809	Acceptable	EPA 6020
1020	Beryllium	mg/kg	72.0	75.8	48.4 - 90.2	Acceptable	EPA 6020
1025	Boron	mg/kg	82.9	127	60.5 - 139	Acceptable	EPA 6020
1030	Cadmium	mg/kg	87.6	88.1	53.7 - 103	Acceptable	EPA 6020
1035	Calcium	mg/kg	4060	4380	2790 - 5370	Acceptable	EPA 6020
1040	Chromium	mg/kg	130	142	90.9 - 167	Acceptable	EPA 6020
1050	Cobalt	mg/kg	125	135	81.5 - 151	Acceptable	EPA 6020
1055	Copper	mg/kg	99.0	102	66.9 - 121	Acceptable	EPA 6020
1070	Iron	mg/kg	10800	15300	620 - 27400	Acceptable	EPA 6020
1075	Lead	mg/kg	108	113	75.7 - 134	Acceptable	EPA 6020
1085	Magnesium	mg/kg	2030	2590	1560 - 3220	Acceptable	EPA 6020
1090	Manganese	mg/kg	324	337	230 - 420	Acceptable	EPA 6020
1095	Mercury	mg/kg	3.59	3.86	2.28 - 5.52	Acceptable	EPA 6020
1100	Molybdenum	mg/kg	175	188	117 - 211	Acceptable	EPA 6020
1105	Nickel	mg/kg	224	237	149 - 273	Acceptable	EPA 6020
1125	Potassium	mg/kg	2000	2450	1400 - 3560	Acceptable	EPA 6020
1140	Selenium	mg/kg	115	116	68.6 - 139	Acceptable	EPA 6020
1150	Silver	mg/kg	168	172	84.6 - 221	Acceptable	EPA 6020
1155	Sodium	mg/kg	735	776	473 - 981	Acceptable	EPA 6020
1160	Strontium	mg/kg	66.5	69.1	44.0 - 92.6	Acceptable	EPA 6020
1165	Thallium	mg/kg	146	149	90.7 - 175	Acceptable	EPA 6020
1175	Tin	mg/kg	127	138	77.8 - 170	Acceptable	EPA 6020
1180	Titanium	mg/kg	175	320	0.00 - 547	Acceptable	EPA 6020
1185	Vanadium	mg/kg	148	165	95.6 - 196	Acceptable	EPA 6020
1190	Zinc	mg/kg	153	164	101 - 199	Acceptable	EPA 6020

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

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Study Dates: 10/24/05 - 12/08/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
PAHs by HPLC in Soil							
5500	Acenaphthene	µg/kg	61.9	152	29.5 - 170	Acceptable	EPA 8310 UV
5505	Acenaphthylene	µg/kg	422	806	0.00 - 806	Acceptable	EPA 8310 UV
5555	Anthracene	µg/kg	< 10	153	0.00 - 153	Acceptable	EPA 8310 UV
5575	Benzo(a)anthracene	µg/kg	36.5	79.7	22.7 - 79.7	Acceptable	EPA 8310 UV
5585	Benzo(b)fluoranthene	µg/kg	60.3	87.9	32.2 - 108	Acceptable	EPA 8310 UV
5600	Benzo(k)fluoranthene	µg/kg	54.7	87.4	26.5 - 100	Acceptable	EPA 8310 UV
5590	Benzo(g,h,i)perylene	µg/kg	45.5	84.3	19.8 - 101	Acceptable	EPA 8310 UV
5580	Benzo(a)pyrene	µg/kg	27.5	156	9.21 - 156	Acceptable	EPA 8310 UV
5855	Chrysene	µg/kg	38.4	57.9	20.8 - 70.0	Acceptable	EPA 8310 UV
5895	Dibenz(a,h)anthracene	µg/kg	61.6	86.8	26.9 - 101	Acceptable	EPA 8310 UV
6265	Fluoranthene	µg/kg	68.2	91.2	31.6 - 117	Acceptable	EPA 8310 UV
6270	Fluorene	µg/kg	94.5	196	60.1 - 206	Acceptable	EPA 8310 UV
6315	Indeno(1,2,3-cd)pyrene	µg/kg	41.3	69.2	22.5 - 82.3	Acceptable	EPA 8310 UV
5005	Naphthalene	µg/kg	71.8	210	0.00 - 315	Acceptable	EPA 8310 UV
6615	Phenanthrene	µg/kg	44.7	62.1	21.7 - 74.9	Acceptable	EPA 8310 UV
6665	Pyrene	µg/kg	64.4	76.3	26.0 - 81.4	Acceptable	EPA 8310 UV
TCLP Metals in Soil							
1005	Antimony	mg/L	1.62	1.64	0.930 - 2.07	Acceptable	EPA 6020
1010	Arsenic	mg/L	1.50	1.51	0.953 - 2.03	Acceptable	EPA 6020
1015	Barium	mg/L	3.08	3.59	2.24 - 3.88	Acceptable	EPA 6020
1020	Beryllium	mg/L	2.57	2.57	1.47 - 3.21	Acceptable	EPA 6020
1030	Cadmium	mg/L	1.67	1.83	1.26 - 2.14	Acceptable	EPA 6020
1040	Chromium	mg/L	2.33	2.81	1.71 - 3.11	Acceptable	EPA 6020
1075	Lead	mg/L	2.40	2.83	1.45 - 3.53	Acceptable	EPA 6020
1095	Mercury	mg/L	0.740	0.523	0.428 - 1.02	Acceptable	EPA 6020
1105	Nickel	mg/L	4.22	4.91	3.37 - 5.47	Acceptable	EPA 6020
1140	Selenium	mg/L	2.81	3.04	1.76 - 3.71	Acceptable	EPA 6020
1150	Silver	mg/L	0.424	0.350	0.0380 - 0.650	Acceptable	EPA 6020
1190	Zinc	mg/L	2.30	3.29	1.49 - 3.49	Acceptable	EPA 6020

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-65 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 EPA ID: KS00902 State ID: E-10254 Report Issued: 02/08/02

InterLaB™ WS Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Values are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998 and the National Environmental Laboratory Accreditation Conference (NELAC) program criteria as applicable. A parameter not added to the standard is given an Assigned Value of "0" per the guidelines contained in the USEPA's Criteria Document and NELAC standards.

The Acceptance Limits are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, the NELAC PT program criteria, state specific PT program requirements or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For a further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside the Acceptance Limits.
No Evaluation	=	Reported Value cannot be evaluated.

The Method Description is the method the laboratory reported to ERA.

InterLaB™ WS Study Discussion:

ERA WatR™ Supply Proficiency Testing Study 65 has been reviewed by ERA Senior Management and certified compliant with the requirements of the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document (December 1998), the NELAC Standards Chapter 2 and Associated Appendices, applicable state PT programs, and those contained in the National Institute of Standards and Technology NVLAP Handbooks 150 and 150-19. ERA is a NIST NVLAP accredited PT Provider (Lab Code 2003860).

The opening date for the WatR™ Supply Proficiency Testing Program WS-65 was December 11, 2001, and the closing date was January 25, 2002.

The WatR™ Supply 65 (WS-65) results were examined for any study anomalies. A full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the WS-65 standards met the acceptance criteria contained in the USEPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, and the National Voluntary Laboratory Accreditation Program, Handbook 150-19 for Chemical Calibration for Providers of Proficiency Testing.

The data submitted by participating laboratories was also examined for study anomalies. There were two anomalies found during the review of the study data. These anomalies are addressed on the next page.

The WatR™ Supply Proficiency Testing Program Study WS-65 report shall not be reproduced except in its entirety and without the full written permission of the participating laboratory. The report must not be used by the participating laboratory to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding WatR™ Supply Study, WS-65, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

Study Discussion Concerning WS-65 Nutrients: ortho-Phosphate as P

During the review of the WS-65 Study data, ERA observed a failure rate of 27.3% for ortho-Phosphate as P in the WS Nutrients standard. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification data for both accuracy and homogeneity. Our review of the data confirmed that the standard meets all requirements contained within the USEPA Criteria Document as well as NIST NVLAP criteria to be judged 'fit for use'.

The acceptance limits for this analyte are calculated using the regression equation found in the USEPA Criteria Document. The average failure rate for ortho-Phosphate as P in ERA's last thirty PT studies is 31.0%. The range of failure rates for these ERA studies is 16.7% to 43.8%. USEPA reported a historical failure rate of 21.8% for five of its WS studies. The range of failure rates for the USEPA studies is not available. The failure rate for ortho-Phosphate as P in ERA's WS-65 Study would be 6.06% using ERA's statistically determined regression equation.

Our review of the acceptance ranges for five of the historical USEPA studies for which we have data indicates that the acceptance limits used historically by USEPA: 1) varied from study to study; and 2) were considerably wider in four out of five studies than is required currently. Therefore, the higher failure rate currently observed is not surprising. ERA will communicate this information on failure rates to USEPA and NELAC. When reviewing each laboratory's performance evaluation for this analyte one must take the above information into consideration.

Study Discussion Concerning WS-65 Hardness: Calcium Hardness as CaCO3

During the review of the WS-65 Study data, ERA observed a failure rate of 35.7% for Calcium Hardness as CaCO3 in the WS Hardness standard. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification data for both accuracy and homogeneity. Our review of the data confirmed that the standard meets all requirements contained within the USEPA Criteria Document as well as NIST NVLAP criteria to be judged 'fit for use'.

The WS-65 Study data indicated a normal distribution around the USEPA estimated mean calculated from ERA's assigned value. The acceptance limits for this analyte are calculated using the regression equation found in the USEPA Criteria Document. The average failure rate for Calcium Hardness as CaCO3 in ERA's last twenty PT studies is 20.4%. The range of failure rates for these ERA studies is 8.33% to 37.0%. USEPA reported a historical failure rate of 15.5% for eight of its WS studies. The range of failure rates for the USEPA studies is not available. The failure rate for Calcium Hardness as CaCO3 in ERA's WS-65 Study would be 21.4% using ERA's statistically determined regression equation.

ERA will communicate this information on failure rates to USEPA and NELAC. When reviewing each laboratory's performance evaluation for this analyte, one must take the above information into consideration.

If you have any further questions please feel free to call ERA's Inorganic Chemistry Department at 1-800-372-0122.

NVLAP® Lab code: 200386-0

ENVIRONMENTAL
RESOURCE ASSOCIATES®

Study: **WS65**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **ANALYTICAL MANAGEMENT LABS**

Report Type: **Complete**

Report Method: **Method A**

5540 Marshall St., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-65 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 02/08/02**
State ID: E-10254
ERA Laboratory Code: A5230-01

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Volatiles							
0039	Benzene †	µg/L	12.07	14.6	11.7 - 17.5	Acceptable	EPA 524.2
0037	Carbon tetrachloride †	µg/L	8.87	11.9	9.52 - 14.3	Not Acceptable	EPA 524.2
0049	Chlorobenzene †	µg/L	18.68	19.6	15.7 - 23.5	Acceptable	EPA 524.2
0054	1,2-Dichlorobenzene †	µg/L	8.98	9.29	5.57 - 13.0	Acceptable	EPA 524.2
0041	1,4-Dichlorobenzene †	µg/L	7.53	7.74	4.64 - 10.8	Acceptable	EPA 524.2
0035	1,2-Dichloroethane †	µg/L	14.61	16.2	13.0 - 19.4	Acceptable	EPA 524.2
0034	1,1-Dichloroethylene †	µg/L	17.32	18.0	14.4 - 21.6	Acceptable	EPA 524.2
0043	cis-1,2-Dichloroethylene †	µg/L	28.36	28.2	22.6 - 33.8	Acceptable	EPA 524.2
0042	trans-1,2-Dichloroethylene †	µg/L	29.72	31.3	25.0 - 37.6	Acceptable	EPA 524.2
0044	1,2-Dichloropropane †	µg/L	13.54	14.9	11.9 - 17.9	Acceptable	EPA 524.2
0048	Ethylbenzene †	µg/L	3.05	3.76	2.26 - 5.26	Acceptable	EPA 524.2
0055	Methylene chloride †	µg/L	9.94	11.2	8.96 - 13.4	Acceptable	EPA 524.2
0053	Styrene †	µg/L	15.06	17.6	14.1 - 21.1	Acceptable	EPA 524.2
0040	Tetrachloroethylene †	µg/L	12.38	16.1	12.9 - 19.3	Not Acceptable	EPA 524.2
0047	Toluene †	µg/L	6.88	8.64	5.18 - 12.1	Acceptable	EPA 524.2
0076	1,2,4-Trichlorobenzene †	µg/L	10.73	12.4	9.92 - 14.9	Acceptable	EPA 524.2
0036	1,1,1-Trichloroethane †	µg/L	5.98	7.68	4.61 - 10.8	Acceptable	EPA 524.2
0061	1,1,2-Trichloroethane †	µg/L	5.62	6.03	3.62 - 8.44	Acceptable	EPA 524.2
0038	Trichloroethylene †	µg/L	9.95	12.9	10.3 - 15.5	Not Acceptable	EPA 524.2
0032	Vinyl chloride †	µg/L	26.43	25.0	15.0 - 35.0	Acceptable	EPA 524.2
0090	Xylenes, total †	µg/L	24.32	27.9	22.3 - 33.5	Acceptable	EPA 524.2
Halomethanes							
0019	Bromodichloromethane †	µg/L	35.46	37.9	30.3 - 45.5	Acceptable	EPA 524.2
0018	Bromoform †	µg/L	16.5	21.5	17.2 - 25.8	Not Acceptable	EPA 524.2
0020	Chlorodibromomethane †	µg/L	4.4	5.02	3.01 - 7.03	Acceptable	EPA 524.2
0017	Chloroform †	µg/L	17.48	16.3	13.0 - 19.6	Acceptable	EPA 524.2
0021	Total Trihalomethanes †	µg/L	73.84	80.7	64.6 - 96.8	Acceptable	EPA 524.2
Unregulated Volatiles							
0067	Bromobenzene †	µg/L	12.2	13.7	11.0 - 16.4	Acceptable	EPA 524.2
0089	Bromochloromethane †	µg/L	6.8	7.19	4.31 - 10.1	Acceptable	EPA 524.2
0069	Bromomethane †	µg/L	< 1	0.00		Acceptable	EPA 524.2
0079	n-Butylbenzene †	µg/L	< 1	0.00		Acceptable	EPA 524.2
0086	sec-Butylbenzene †	µg/L	2.36	3.31	1.99 - 4.63	Acceptable	EPA 524.2
0085	tert-Butylbenzene †	µg/L	12.5	15.7	12.6 - 18.8	Not Acceptable	EPA 524.2
0267	tert-Butylmethyl ether (MTBE)	µg/L	< 1	0.00		Acceptable	EPA 524.2
0070	Chloroethane †	µg/L	< 1	0.00		Acceptable	EPA 524.2
0068	Chloromethane †	µg/L	< 1	0.00		Acceptable	EPA 524.2

All analytes are included in ERA's A2LA accredidation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accredidation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-65 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 02/08/02**
State ID: E-10254
ERA Laboratory Code: A5230-01

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Unregulated Volatiles (Cont.)							
0071	2-Chlorotoluene †	µg/L	4.1	3.89	2.33 - 5.45	Acceptable	EPA 524.2
0072	4-Chlorotoluene †	µg/L	7.13	8.83	5.30 - 12.4	Acceptable	EPA 524.2
0057	Dibromomethane †	µg/L	15.36	17.1	13.7 - 20.5	Acceptable	EPA 524.2
0066	1,3-Dichlorobenzene †	µg/L	13.26	14.9	11.9 - 17.9	Acceptable	EPA 524.2
0088	Dichlorodifluoromethane †	µg/L	< 1	0.00		Acceptable	EPA 524.2
0056	1,1-Dichloroethane †	µg/L	16.38	18.5	14.8 - 22.2	Acceptable	EPA 524.2
0059	1,3-Dichloropropane †	µg/L	8.22	9.90	5.94 - 13.9	Acceptable	EPA 524.2
0060	2,2-Dichloropropane †	µg/L	3.63	5.93	3.56 - 8.30	Acceptable	EPA 524.2
0058	1,1-Dichloropropene †	µg/L	8.82	12.1	9.68 - 14.5	Not Acceptable	EPA 524.2
0152	cis-1,3-Dichloropropene †	µg/L	< 1	0.00		Acceptable	EPA 524.2
0153	trans-1,3-Dichloropropene †	µg/L	< 1	0.00		Acceptable	EPA 524.2
0087	Fluorotrichloromethane †	µg/L		0.00			
0081	Hexachlorobutadiene †	µg/L	12.42	15.8	12.6 - 19.0	Not Acceptable	EPA 524.2
0084	Isopropylbenzene †	µg/L	1.86	2.99	1.79 - 4.19	Acceptable	EPA 524.2
0083	4-Isopropyltoluene †	µg/L	9.76	12.6	10.1 - 15.1	Not Acceptable	EPA 524.2
0078	n-Propylbenzene †	µg/L	7.62	10.3	8.24 - 12.4	Not Acceptable	EPA 524.2
0063	1,1,1,2-Tetrachloroethane †	µg/L	4.94	5.89	3.53 - 8.25	Acceptable	EPA 524.2
0065	1,1,2,2-Tetrachloroethane †	µg/L	11.78	13.2	10.6 - 15.8	Acceptable	EPA 524.2
0077	1,2,3-Trichlorobenzene †	µg/L	16.1	15.8	12.6 - 19.0	Acceptable	EPA 524.2
0064	1,2,3-Trichloropropane †	µg/L	5.33	7.06	4.24 - 9.88	Acceptable	EPA 524.2
0075	1,2,4-Trimethylbenzene †	µg/L	14.47	16.9	13.5 - 20.3	Acceptable	EPA 524.2
0082	1,3,5-Trimethylbenzene †	µg/L	13.2	16.6	13.3 - 19.9	Not Acceptable	EPA 524.2
EDB/DBCP							
0045	Dibromochloropropane (DBCP) †	µg/L	1.467	1.59	0.954 - 2.23	Acceptable	EPA 504
0046	Ethylene Dibromide (EDB) †	µg/L	0.871	0.948	0.569 - 1.33	Acceptable	EPA 504
9807	1,2,3-Trichloropropane	µg/L		0.00			
Regulated Semi - Voas #1							
0278	Acenaphthene	µg/L	< 0.2	0.00		Acceptable	EPA 525
0119	Acenaphthylene	µg/L	< 0.2	0.00		Acceptable	EPA 525
0120	Anthracene	µg/L	< 0.2	0.00		Acceptable	EPA 525
0121	Benzo(a)anthracene	µg/L	< 0.2	0.00		Acceptable	EPA 525
0123	Benzo(b)fluoranthene	µg/L	< 0.2	0.00		Acceptable	EPA 525
0124	Benzo(k)fluoranthene	µg/L	< 0.2	0.00		Acceptable	EPA 525
0125	Benzo(g,h,i)perylene	µg/L	16.5	15.4	7.70 - 23.1	Acceptable	EPA 525
0122	Benzo(a)pyrene †	µg/L	1.73	1.46	0.350 - 1.89	Acceptable	EPA 525
0135	Butylbenzylphthalate	µg/L	28.84	23.4	7.02 - 39.8	Acceptable	EPA 525
0126	Chrysene	µg/L	< 0.2	0.00		Acceptable	EPA 525

All analytes are included in ERA's A2LA accredidation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accredidation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-65 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902 **Report Issued: 02/08/02**
State ID: E-10254
ERA Laboratory Code: A5230-01

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Semi - Voas #1 (Cont.)							
0127	Dibenz(a,h)anthracene	µg/L	< 0.2	0.00		Acceptable	EPA 525
0149	Di-n-butylphthalate	µg/L	< 0.2	0.00		Acceptable	EPA 525
0150	Diethylphthalate	µg/L	< 0.2	0.00		Acceptable	EPA 525
0151	Dimethylphthalate	µg/L	< 0.2	0.00		Acceptable	EPA 525
0274	Di-n-octylphthalate	µg/L	< 0.2	0.00		Acceptable	EPA 525
0134	bis(2-Ethylhexyl)adipate †	µg/L	33.43	32.5	13.1 - 47.0	Acceptable	EPA 525
0136	bis(2-Ethylhexyl)phthalate †	µg/L	26.57	20.8	8.55 - 32.2	Acceptable	EPA 525
0132	Fluoranthene	µg/L	< 0.2	0.00		Acceptable	EPA 525
0128	Fluorene	µg/L	< 0.2	0.00		Acceptable	EPA 525
0129	Indeno(1,2,3-cd)pyrene	µg/L	< 0.2	0.00		Acceptable	EPA 525
0276	1-Methylnaphthalene	µg/L		0.00			
0277	2-Methylnaphthalene	µg/L		0.00			
0133	Naphthalene	µg/L	< 0.2	0.00		Acceptable	EPA 525
0130	Phenanthrene	µg/L	< 0.2	0.00		Acceptable	EPA 525
0131	Pyrene	µg/L	19.91	19.8	9.90 - 29.7	Acceptable	EPA 525

All analytes are included in ERA's A2LA accredidation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accredidation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

WS-66 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 EPA ID: KS00902 State ID: E-10254 Report Issued: 03/18/02

InterLaB™ WS Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Values are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998 and the National Environmental Laboratory Accreditation Conference (NELAC) program criteria as applicable. A parameter not added to the standard is given an Assigned Value of "0" per the guidelines contained in the USEPA's Criteria Document and NELAC standards.

The Acceptance Limits are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, the NELAC PT program criteria, state specific PT program requirements or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For a further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside of the Acceptance Limits.
No Evaluation	=	Reported Value cannot be evaluated.

The Method Description is the method the laboratory reported to ERA.

InterLaB™ WS Study Discussion:

ERA WatR™ Supply Proficiency Testing Study 66 has been reviewed by ERA Senior Management and certified compliant with the requirements of the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document (December 1998), the NELAC Standards Chapter 2 and Associated Appendices, applicable state PT programs, and those contained in the National Institute of Standards and Technology NVLAP Handbooks 150 and 150-19. ERA is a NIST NVLAP accredited PT Provider (Lab Code 2003860).

The opening date for the WatR™ Supply Proficiency Testing Program WS-66 was January 14, 2002, and the closing date was February 28, 2002.

The WatR™ Supply 66 (WS-66) results were examined for any study anomalies. A full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the WS-66 standards met the acceptance criteria contained in the USEPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, and the National Voluntary Laboratory Accreditation Program, Handbook 150-19 for Chemical Calibration for Providers of Proficiency Testing.

The data submitted by participating laboratories was also examined for study anomalies. There was one anomaly found during the review of the study data. This anomaly is addressed on the next page.

The WatR™ Supply Proficiency Testing Program Study WS-66 report shall not be reproduced except in its entirety and without the full written permission of the participating laboratory. The report must not be used by the participating laboratory to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding WatR™ Supply Study, WS-66, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

Study Discussion Concerning WS-66 Nutrients:
ortho-Phosphate as P

During the review of the WS-66 Study data, ERA observed a failure rate of 42.9% for ortho-Phosphate as P in the WS Nutrients standard. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification data for both accuracy and homogeneity. Our review of the data confirmed that the standard meets all requirements contained within the USEPA Criteria Document as well as NIST NVLAP criteria to be judged 'fit for use'.

The acceptance limits for this analyte are calculated using the regression equation found in the USEPA Criteria Document. The average failure rate for ortho-Phosphate as P in ERA's last twenty-three PT studies is 31.8%. The range of failure rates for these ERA studies is 16.7% to 43.8%. USEPA reported a historical failure rate of 21.8% for five of its WS studies. The range of failure rates for the USEPA studies is not available. The failure rate for ortho-Phosphate as P in ERA's WS-66 Study would be 17.1% using ERA's statistically determined regression equation.

Our review of the acceptance ranges for five of the historical USEPA studies for which we have data indicates that the acceptance limits used historically by USEPA: 1) varied from study to study; and 2) were considerably wider in four out of five studies than is required currently. Therefore, the higher failure rate currently observed is not surprising. ERA will communicate this information on failure rates to USEPA and NELAC. When reviewing each laboratory's performance evaluation for this analyte one must take the above information into consideration.

Study Discussion Concerning WS-66: Method Codes

Currently, due to the variety of ways methods are being provided to ERA by the participating laboratories for WS-66, methods codes are currently not being used. Once the entry of method descriptions has been standardized, ERA will be providing method codes, both in the hard copy and electronic versions of the final report.

NVLAP® Lab code: 200386-0

ENVIRONMENTAL
RESOURCE ASSOCIATES®

Study: **WS66**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **ANALYTICAL MANAGEMENT LABS**

Report Type: **Complete**

Report Method: **Method A**

5540 Marshall St., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-66 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902
State ID: E-10254
ERA Laboratory Code: A5230-01
Report Issued: 03/18/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Metals							
0235	Aluminum	µg/L	853	900	809 - 987	Acceptable	200.8
0140	Antimony †	µg/L	17.2	17.0	11.9 - 22.1	Acceptable	200.8
0001	Arsenic †	µg/L	40.1	40.8	35.6 - 45.6	Acceptable	200.8
0002	Barium †	µg/L	1334	1380	1170 - 1590	Acceptable	200.8
0141	Beryllium †	µg/L	4.18	4.29	3.65 - 4.93	Acceptable	200.8
0226	Boron †	µg/L		488	460 - 532		
0003	Cadmium †	µg/L	25.0	25.7	20.6 - 30.8	Acceptable	200.8
0004	Chromium †	µg/L	79.5	78.4	66.6 - 90.2	Acceptable	200.8
0091	Copper †	µg/L	270	276	248 - 304	Acceptable	200.8
0284	Iron	µg/L	1756	1830	1680 - 1980	Acceptable	200.8
0005	Lead †	µg/L	28.4	29.2	20.4 - 38.0	Acceptable	200.8
0236	Manganese †	µg/L	335	336	312 - 354	Acceptable	200.8
0237	Molybdenum †	µg/L	44.3	43.9	36.7 - 50.9	Acceptable	200.8
0142	Nickel †	µg/L	290	288	245 - 331	Acceptable	200.8
0007	Selenium †	µg/L	48.7	50.4	40.3 - 60.5	Acceptable	200.8
0008	Silver	µg/L	353	337	305 - 370	Acceptable	200.8
0143	Thallium †	µg/L	3.98	4.11	2.88 - 5.34	Acceptable	200.8
0238	Vanadium	µg/L	419	428	398 - 455	Acceptable	200.8
0239	Zinc †	µg/L	895	868	797 - 933	Acceptable	200.8
Mercury							
0006	Mercury †	µg/L	4.84	5.14	3.60 - 6.68	Acceptable	200.8

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

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ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902
State ID: E-10254
ERA Laboratory Code: A5230-01
Report Issued: 03/18/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Volatiles							
0039	Benzene †	µg/L	13.31	12.9	10.3 - 15.5	Acceptable	EPA 524.2
0037	Carbon tetrachloride †	µg/L	7.22	6.79	4.07 - 9.51	Acceptable	EPA 524.2
0049	Chlorobenzene †	µg/L	17.24	16.5	13.2 - 19.8	Acceptable	EPA 524.2
0054	1,2-Dichlorobenzene †	µg/L	15.65	14.4	11.5 - 17.3	Acceptable	EPA 524.2
0041	1,4-Dichlorobenzene †	µg/L	14.65	14.0	11.2 - 16.8	Acceptable	EPA 524.2
0035	1,2-Dichloroethane †	µg/L	7.18	6.71	4.03 - 9.39	Acceptable	EPA 524.2
0034	1,1-Dichloroethylene †	µg/L	15.6	16.9	13.5 - 20.3	Acceptable	EPA 524.2
0043	cis-1,2-Dichloroethylene †	µg/L	41.94	39.5	31.6 - 47.4	Acceptable	EPA 524.2
0042	trans-1,2-Dichloroethylene †	µg/L	28.95	29.2	23.4 - 35.0	Acceptable	EPA 524.2
0044	1,2-Dichloropropane †	µg/L	13.95	12.9	10.3 - 15.5	Acceptable	EPA 524.2
0048	Ethylbenzene †	µg/L	18.03	17.5	14.0 - 21.0	Acceptable	EPA 524.2
0055	Methylene chloride †	µg/L	16.73	18.9	15.1 - 22.7	Acceptable	EPA 524.2
0053	Styrene †	µg/L	6.59	6.08	3.65 - 8.51	Acceptable	EPA 524.2
0040	Tetrachloroethylene †	µg/L	14.86	14.6	11.7 - 17.5	Acceptable	EPA 524.2
0047	Toluene †	µg/L	16.66	16.5	13.2 - 19.8	Acceptable	EPA 524.2
0076	1,2,4-Trichlorobenzene †	µg/L	6.71	6.45	3.87 - 9.03	Acceptable	EPA 524.2
0036	1,1,1-Trichloroethane †	µg/L	11.89	11.6	9.28 - 13.9	Acceptable	EPA 524.2
0061	1,1,2-Trichloroethane †	µg/L	4.13	3.98	2.39 - 5.57	Acceptable	EPA 524.2
0038	Trichloroethylene †	µg/L	9.93	9.90	5.94 - 13.9	Acceptable	EPA 524.2
0032	Vinyl chloride †	µg/L	22.93	21.0	12.6 - 29.4	Acceptable	EPA 524.2
0090	Xylenes, total †	µg/L	31.13	30.1	24.1 - 36.1	Acceptable	EPA 524.2
Halomethanes							
0019	Bromodichloromethane †	µg/L	50.90	45.4	36.3 - 54.5	Acceptable	EPA 524.2
0018	Bromoform †	µg/L	43.13	34.9	27.9 - 41.9	Not Acceptable	EPA 524.2
0020	Chlorodibromomethane †	µg/L	8.12	7.11	4.27 - 9.95	Acceptable	EPA 524.2
0017	Chloroform †	µg/L	4.85	4.20	2.52 - 5.88	Acceptable	EPA 524.2
0021	Total Trihalomethanes †	µg/L	107.0	91.6	73.3 - 110	Acceptable	EPA 524.2
Unregulated Volatiles							
0067	Bromobenzene †	µg/L	10.06	8.74	5.24 - 12.2	Acceptable	EPA 524.2
0089	Bromochloromethane †	µg/L	19.9	18.5	14.8 - 22.2	Acceptable	EPA 524.2
0069	Bromomethane †	µg/L	< 1	0.00		Acceptable	EPA 524.2
0079	n-Butylbenzene †	µg/L	< 1	0.00		Acceptable	EPA 524.2
0086	sec-Butylbenzene †	µg/L	14.44	17.2	13.8 - 20.6	Acceptable	EPA 524.2
0085	tert-Butylbenzene †	µg/L	5.94	6.37	3.82 - 8.92	Acceptable	EPA 524.2
0267	tert-Butylmethyl ether (MTBE)	µg/L	< 1	0.00		Acceptable	EPA 524.2
0070	Chloroethane †	µg/L	< 1	0.00		Acceptable	EPA 524.2
0068	Chloromethane †	µg/L	< 1	0.00		Acceptable	EPA 524.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-66 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902
State ID: E-10254
ERA Laboratory Code: A5230-01
Report Issued: 03/18/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Unregulated Volatiles (Cont.)							
0071	2-Chlorotoluene †	µg/L	11.34	14.2	11.4 - 17.0	Not Acceptable	EPA 524.2
0072	4-Chlorotoluene †	µg/L	2.69	3.04	1.82 - 4.26	Acceptable	EPA 524.2
0057	Dibromomethane †	µg/L	13.99	12.3	9.84 - 14.8	Acceptable	EPA 524.2
0066	1,3-Dichlorobenzene †	µg/L	6.61	7.52	4.51 - 10.5	Acceptable	EPA 524.2
0088	Dichlorodifluoromethane †	µg/L	< 1	0.00		Acceptable	EPA 524.2
0056	1,1-Dichloroethane †	µg/L	17.36	16.5	13.2 - 19.8	Acceptable	EPA 524.2
0059	1,3-Dichloropropane †	µg/L	6.49	7.42	4.45 - 10.4	Acceptable	EPA 524.2
0060	2,2-Dichloropropane †	µg/L	4.98	6.67	4.00 - 9.34	Acceptable	EPA 524.2
0058	1,1-Dichloropropene †	µg/L	6.94	7.22	4.33 - 10.1	Acceptable	EPA 524.2
0152	cis-1,3-Dichloropropene †	µg/L	< 1	0.00		Acceptable	EPA 524.2
0153	trans-1,3-Dichloropropene †	µg/L	< 1	0.00		Acceptable	EPA 524.2
0087	Fluorotrichloromethane †	µg/L		0.00			
0081	Hexachlorobutadiene †	µg/L	18.41	17.2	13.8 - 20.6	Acceptable	EPA 524.2
0084	Isopropylbenzene †	µg/L	8.22	9.64	5.78 - 13.5	Acceptable	EPA 524.2
0083	4-Isopropyltoluene †	µg/L	8.95	9.43	5.66 - 13.2	Acceptable	EPA 524.2
0078	n-Propylbenzene †	µg/L	6.65	6.98	4.19 - 9.77	Acceptable	EPA 524.2
0063	1,1,1,2-Tetrachloroethane †	µg/L	4.66	4.10	2.46 - 5.74	Acceptable	EPA 524.2
0065	1,1,2,2-Tetrachloroethane †	µg/L	5.79	4.94	2.96 - 6.92	Acceptable	EPA 524.2
0077	1,2,3-Trichlorobenzene †	µg/L	< 1	0.00		Acceptable	EPA 524.2
0064	1,2,3-Trichloropropane †	µg/L	8.75	9.46	5.68 - 13.2	Acceptable	EPA 524.2
0075	1,2,4-Trimethylbenzene †	µg/L	14.55	14.7	11.8 - 17.6	Acceptable	EPA 524.2
0082	1,3,5-Trimethylbenzene †	µg/L	7.26	7.78	4.67 - 10.9	Acceptable	EPA 524.2
EDB/DBCP							
0045	Dibromochloropropane (DBCP) †	µg/L	1.40	1.53	0.918 - 2.14	Acceptable	EPA 504
0046	Ethylene Dibromide (EDB) †	µg/L	1.53	1.68	1.01 - 2.35	Acceptable	EPA 504
9807	1,2,3-Trichloropropane	ug/L		0.563	0.338 - 0.788		
Regulated Semi - Voas #1							
0278	Acenaphthene	µg/L	< 0.4	0.00		Acceptable	EPA 525
0119	Acenaphthylene	µg/L	< 0.4	0.00		Acceptable	EPA 525
0120	Anthracene	µg/L	< 0.4	0.00		Acceptable	EPA 525
0121	Benzo(a)anthracene	µg/L	< 0.4	0.00		Acceptable	EPA 525
0123	Benzo(b)fluoranthene	µg/L	14.68	14.9	7.45 - 22.4	Acceptable	EPA 525
0124	Benzo(k)fluoranthene	µg/L	20.64	23.5	11.8 - 35.3	Acceptable	EPA 525
0125	Benzo(g,h,i)perylene	µg/L	< 0.4	0.00		Acceptable	EPA 525
0122	Benzo(a)pyrene †	µg/L	0.47	0.906	0.228 - 1.19	Acceptable	EPA 525
0135	Butylbenzylphthalate	µg/L	2.72	0.00		Not Acceptable	EPA 525
0126	Chrysene	µg/L	< 0.4	0.00		Acceptable	EPA 525

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902
State ID: E-10254
ERA Laboratory Code: A5230-01
Report Issued: 03/18/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Semi - Voas #1 (Cont.)							
0127	Dibenz(a,h)anthracene	µg/L	< 0.4	0.00		Acceptable	EPA 525
0149	Di-n-butylphthalate	µg/L	< 0.4	0.00		Acceptable	EPA 525
0150	Diethylphthalate	µg/L	38.74	39.2	11.8 - 66.6	Acceptable	EPA 525
0151	Dimethylphthalate	µg/L	< 0.4	0.00		Acceptable	EPA 525
0274	Di-n-octylphthalate	µg/L	< 0.4	0.00		Acceptable	EPA 525
0134	bis(2-Ethylhexyl)adipate †	µg/L	49.74	41.5	17.3 - 59.8	Acceptable	EPA 525
0136	bis(2-Ethylhexyl)phthalate †	µg/L	14.16	11.5	4.33 - 17.6	Acceptable	EPA 525
0132	Fluoranthene	µg/L	< 0.4	0.00		Acceptable	EPA 525
0128	Fluorene	µg/L	18.33	24.7	12.4 - 37.1	Acceptable	EPA 525
0129	Indeno(1,2,3-cd)pyrene	µg/L	< 0.4	0.00		Acceptable	EPA 525
0276	1-Methylnaphthalene	µg/L		0.00			
0277	2-Methylnaphthalene	µg/L		0.00			
0133	Naphthalene	µg/L	< 0.4	0.00		Acceptable	EPA 525
0130	Phenanthrene	µg/L	< 0.4	0.00		Acceptable	EPA 525
0131	Pyrene	µg/L	< 0.4	0.00		Acceptable	EPA 525

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

InterLaB™ WS Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Values are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998 and the National Environmental Laboratory Accreditation Conference (NELAC) program criteria as applicable. A parameter not added to the standard is given an Assigned Value of "0" per the guidelines contained in the USEPA's Criteria Document and NELAC standards.

The Acceptance Limits are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, the NELAC PT program criteria, state specific PT program requirements or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For a further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside of the Acceptance Limits.
No Evaluation	=	Reported Value cannot be evaluated.

The Method Description is the method the laboratory reported to ERA.

InterLaB™ WS Study Discussion:

ERA WatR™ Supply Proficiency Testing Study 67 has been reviewed by ERA Senior Management and certified compliant with the requirements of the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document (December 1998), the NELAC Standards Chapter 2 and Associated Appendices, applicable state PT programs, and those contained in the National Institute of Standards and Technology NVLAP Handbooks 150 and 150-19. ERA is a NIST NVLAP accredited PT Provider (Lab Code 2003860).

The opening date for the WatR™ Supply Proficiency Testing Program WS-67 was February 11, 2002, and the closing date was March 28, 2002.

The WatR™ Supply 67 (WS-67) results were examined for any study anomalies. A full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the WS-67 standards met the acceptance criteria contained in the USEPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, and the National Voluntary Laboratory Accreditation Program, Handbook 150-19 for Chemical Calibration for Providers of Proficiency Testing.

The data submitted by participating laboratories was also examined for study anomalies. There was one anomaly found during the review of the study data. This anomaly is addressed on the next page.

The WatR™ Supply Proficiency Testing Program Study WS-67 report shall not be reproduced except in its entirety and without the full written permission of the participating laboratory. The report must not be used by the participating laboratory to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding WatR™ Supply Study, WS-67, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

Study Discussion Concerning WS-67 Nutrients:
ortho-Phosphate as P

During the review of the WS-67 Study data, ERA observed a failure rate of 25.0% for ortho-Phosphate as P in the WS Nutrients standard. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification data for both accuracy and homogeneity. Our review of the data confirmed that the standard meets all requirements contained within the USEPA Criteria Document as well as NIST NVLAP criteria to be judged 'fit for use'.

The acceptance limits for this analyte are calculated using the regression equation found in the USEPA Criteria Document. The average failure rate for ortho-Phosphate as P in ERA's last thirty-one PT studies is 30.9%. The range of failure rates for these ERA studies is 16.7% to 43.8%. USEPA reported a historical failure rate of 21.8% for five of its WS studies. The range of failure rates for the USEPA studies is not available. The failure rate for ortho-Phosphate as P in ERA's WS-67 Study would be 10.0% using ERA's statistically determined regression equation.

Our review of the acceptance ranges for five of the historical USEPA studies for which we have data indicates that the acceptance limits used historically by USEPA: 1) varied from study to study; and 2) were considerably wider in four out of five studies than is required currently. Therefore, the higher failure rate currently observed is not surprising. ERA will communicate this information on failure rates to USEPA and NELAC. When reviewing each laboratory's performance evaluation for this analyte one must take the above information into consideration.

Study Discussion Concerning WS-67: Method Codes

Currently, due to the variety of ways methods are being provided to ERA by the participating laboratories for WS-67, methods codes are currently not being used. Once the entry of method descriptions has been standardized, ERA will be providing method codes, both in the hard copy and electronic versions of the final report.

ENVIRONMENTAL
RESOURCE ASSOCIATES®

Study: **WS67**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **ANALYTICAL MANAGEMENT LABS**

Report Type: **Complete**

Report Method: **Method A**

5540 Marshall St., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-67 Final Complete Report

ANALYTICAL MANAGEMENT LABS
15130 B South Keeler
Olathe, Ks 66062

EPA ID: KS00902
State ID:
ERA Laboratory Code: A5230-01
Report Issued: 04/15/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Metals							
0235	Aluminum	µg/L	399	417	370 - 465	Acceptable	200.8
0140	Antimony †	µg/L	21.6	21.3	14.9 - 27.7	Acceptable	200.8
0001	Arsenic †	µg/L	78.1	82.1	71.8 - 91.7	Acceptable	200.8
0002	Barium †	µg/L	1894	1940	1650 - 2230	Acceptable	200.8
0141	Beryllium †	µg/L	5.85	5.75	4.89 - 6.61	Acceptable	200.8
0226	Boron †	µg/L		474	447 - 517		
0003	Cadmium †	µg/L	9.12	9.37	7.50 - 11.2	Acceptable	200.8
0004	Chromium †	µg/L	123	122	104 - 140	Acceptable	200.8
0091	Copper †	µg/L	379	391	352 - 430	Acceptable	200.8
0284	Iron	µg/L	585	623	572 - 677	Acceptable	200.8
0005	Lead †	µg/L	35.0	36.0	25.2 - 46.8	Acceptable	200.8
0236	Manganese †	µg/L	106	108	99.2 - 114	Acceptable	200.8
0237	Molybdenum †	µg/L	89.6	89.2	75.0 - 103	Acceptable	200.8
0142	Nickel †	µg/L	360	360	306 - 414	Acceptable	200.8
0007	Selenium †	µg/L	55.7	59.0	47.2 - 70.8	Acceptable	200.8
0008	Silver	µg/L	85.3	71.1	63.9 - 78.1	Not Acceptable	200.8
0143	Thallium †	µg/L	4.49	4.62	3.23 - 6.01	Acceptable	200.8
0238	Vanadium	µg/L	357	375	348 - 399	Acceptable	200.8
0239	Zinc †	µg/L	516	492	450 - 531	Acceptable	200.8
Mercury							
0006	Mercury †	µg/L	1.22	1.08	0.756 - 1.40	Acceptable	200.8

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-67 Final Complete Report

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15130 B South Keeler
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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Volatiles							
0039	Benzene †	µg/L	4.30	4.31	2.59 - 6.03	Acceptable	524.2
0037	Carbon tetrachloride †	µg/L	17.0	16.2	13.0 - 19.4	Acceptable	524.2
0049	Chlorobenzene †	µg/L	9.94	9.63	5.78 - 13.5	Acceptable	524.2
0054	1,2-Dichlorobenzene †	µg/L	8.20	7.74	4.64 - 10.8	Acceptable	524.2
0041	1,4-Dichlorobenzene †	µg/L	12.1	11.6	9.28 - 13.9	Acceptable	524.2
0035	1,2-Dichloroethane †	µg/L	8.66	8.94	5.36 - 12.5	Acceptable	524.2
0034	1,1-Dichloroethylene †	µg/L	9.63	9.01	5.41 - 12.6	Acceptable	524.2
0043	cis-1,2-Dichloroethylene †	µg/L	26.2	24.4	19.5 - 29.3	Acceptable	524.2
0042	trans-1,2-Dichloroethylene †	µg/L	18.8	18.8	15.0 - 22.6	Acceptable	524.2
0044	1,2-Dichloropropane †	µg/L	10.2	9.61	5.77 - 13.5	Acceptable	524.2
0048	Ethylbenzene †	µg/L	7.14	7.02	4.21 - 9.83	Acceptable	524.2
0055	Methylene chloride †	µg/L	6.96	7.44	4.46 - 10.4	Acceptable	524.2
0053	Styrene †	µg/L	5.24	5.00	3.00 - 7.00	Acceptable	524.2
0040	Tetrachloroethylene †	µg/L	9.96	9.72	5.83 - 13.6	Acceptable	524.2
0047	Toluene †	µg/L	12.4	12.3	9.84 - 14.8	Acceptable	524.2
0076	1,2,4-Trichlorobenzene †	µg/L	9.57	9.47	5.68 - 13.3	Acceptable	524.2
0036	1,1,1-Trichloroethane †	µg/L	15.0	14.8	11.8 - 17.8	Acceptable	524.2
0061	1,1,2-Trichloroethane †	µg/L	7.91	7.86	4.72 - 11.0	Acceptable	524.2
0038	Trichloroethylene †	µg/L	12.7	13.5	10.8 - 16.2	Acceptable	524.2
0032	Vinyl chloride †	µg/L	16.5	16.7	10.0 - 23.4	Acceptable	524.2
0090	Xylenes, total †	µg/L	24.0	23.6	18.9 - 28.3	Acceptable	524.2
Halomethanes							
0019	Bromodichloromethane †	µg/L	16.6	15.1	12.1 - 18.1	Acceptable	524.2
0018	Bromoform †	µg/L	12.2	9.78	5.87 - 13.7	Acceptable	524.2
0020	Chlorodibromomethane †	µg/L	23.6	21.7	17.4 - 26.0	Acceptable	524.2
0017	Chloroform †	µg/L	17.8	17.4	13.9 - 20.9	Acceptable	524.2
0021	Total Trihalomethanes †	µg/L	70.2	64.0	51.2 - 76.8	Acceptable	524.2
Unregulated Volatiles							
0067	Bromobenzene †	µg/L	15.2	15.2	12.2 - 18.2	Acceptable	524.2
0089	Bromochloromethane †	µg/L	5.66	5.81	3.49 - 8.13	Acceptable	524.2
0069	Bromomethane †	µg/L	< 0.5	0.00		Acceptable	524.2
0079	n-Butylbenzene †	µg/L	< 0.5	0.00		Acceptable	524.2
0086	sec-Butylbenzene †	µg/L	5.47	5.51	3.31 - 7.71	Acceptable	524.2
0085	tert-Butylbenzene †	µg/L	18.9	17.4	13.9 - 20.9	Acceptable	524.2
0267	tert-Butylmethyl ether (MTBE)	µg/L	< 0.5	0.00		Acceptable	524.2
0070	Chloroethane †	µg/L	< 0.5	0.00		Acceptable	524.2
0068	Chloromethane †	µg/L	< 0.5	0.00		Acceptable	524.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

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EPA ID: KS00902
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ERA Laboratory Code: A5230-01
Report Issued: 04/15/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Unregulated Volatiles (Cont.)							
0071	2-Chlorotoluene †	µg/L	6.44	7.16	4.30 - 10.0	Acceptable	524.2
0072	4-Chlorotoluene †	µg/L	14.5	14.4	11.5 - 17.3	Acceptable	524.2
0057	Dibromomethane †	µg/L	9.34	9.42	5.65 - 13.2	Acceptable	524.2
0066	1,3-Dichlorobenzene †	µg/L	10.8	10.4	8.32 - 12.5	Acceptable	524.2
0088	Dichlorodifluoromethane †	µg/L	< 0.5	0.00		Acceptable	524.2
0056	1,1-Dichloroethane †	µg/L	3.66	3.81	2.29 - 5.33	Acceptable	524.2
0059	1,3-Dichloropropane †	µg/L	14.5	14.5	11.6 - 17.4	Acceptable	524.2
0060	2,2-Dichloropropane †	µg/L	10.7	12.6	10.1 - 15.1	Acceptable	524.2
0058	1,1-Dichloropropene †	µg/L	10.6	9.31	5.59 - 13.0	Acceptable	524.2
0152	cis-1,3-Dichloropropene †	µg/L	< 0.5	0.00		Acceptable	524.2
0153	trans-1,3-Dichloropropene †	µg/L	< 0.5	0.00		Acceptable	524.2
0087	Fluorotrichloromethane †	µg/L		16.7	13.4 - 20.0		
0081	Hexachlorobutadiene †	µg/L	13.2	10.5	8.40 - 12.6	Not Acceptable	524.2
0084	Isopropylbenzene †	µg/L	14.5	14.2	11.4 - 17.0	Acceptable	524.2
0083	4-Isopropyltoluene †	µg/L	4.60	4.07	2.44 - 5.70	Acceptable	524.2
0078	n-Propylbenzene †	µg/L	12.8	11.2	8.96 - 13.4	Acceptable	524.2
0063	1,1,1,2-Tetrachloroethane †	µg/L	7.99	8.08	4.85 - 11.3	Acceptable	524.2
0065	1,1,2,2-Tetrachloroethane †	µg/L	6.04	8.24	4.94 - 11.5	Acceptable	524.2
0077	1,2,3-Trichlorobenzene †	µg/L	11.2	10.5	8.40 - 12.6	Acceptable	524.2
0064	1,2,3-Trichloropropane †	µg/L	2.39	2.97	1.78 - 4.16	Acceptable	524.2
0075	1,2,4-Trimethylbenzene †	µg/L	7.43	6.36	3.82 - 8.90	Acceptable	524.2
0082	1,3,5-Trimethylbenzene †	µg/L	13.1	11.5	9.20 - 13.8	Acceptable	524.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

Study: **WS67**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **ANALYTICAL MANAGEMENT LABS**

Report Type: **Complete**

Report Method: **Method B**

5540 Marshall St., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

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EPA ID: KS00902
State ID:
ERA Laboratory Code: A5230-01
Report Issued: 04/15/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Metals							
0235	Aluminum	µg/L	440	417	370 - 465	Acceptable	200.7
0140	Antimony †	µg/L		21.3	14.9 - 27.7		
0001	Arsenic †	µg/L		82.1	71.8 - 91.7		
0002	Barium †	µg/L	1868	1940	1650 - 2230	Acceptable	200.7
0141	Beryllium †	µg/L	5.38	5.75	4.89 - 6.61	Acceptable	200.7
0226	Boron †	µg/L		474	447 - 517		
0003	Cadmium †	µg/L	8.25	9.37	7.50 - 11.2	Acceptable	200.7
0004	Chromium †	µg/L	129	122	104 - 140	Acceptable	200.7
0091	Copper †	µg/L	377	391	352 - 430	Acceptable	200.7
0284	Iron	µg/L	627	623	572 - 677	Acceptable	200.7
0005	Lead †	µg/L		36.0	25.2 - 46.8		
0236	Manganese †	µg/L	108	108	99.2 - 114	Acceptable	200.7
0237	Molybdenum †	µg/L	92.2	89.2	75.0 - 103	Acceptable	200.7
0142	Nickel †	µg/L	388	360	306 - 414	Acceptable	200.7
0007	Selenium †	µg/L		59.0	47.2 - 70.8		
0008	Silver	µg/L	79.0	71.1	63.9 - 78.1	Not Acceptable	200.7
0143	Thallium †	µg/L		4.62	3.23 - 6.01		
0238	Vanadium	µg/L	364	375	348 - 399	Acceptable	200.7
0239	Zinc †	µg/L	506	492	450 - 531	Acceptable	200.7
Mercury							
0006	Mercury †	µg/L	1.15	1.08	0.756 - 1.40	Acceptable	245.1

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP®

WS-75 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 **EPA ID: KS00902** **Report Issued: 12/17/02**

WS Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Values are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, and the National Environmental Laboratory Accreditation Conference (NELAC) program criteria as applicable. A parameter not added to the standard is given an Assigned Value of "0" per the guidelines contained in the USEPA's Criteria Document and NELAC standards.

The Acceptance Limits are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, the NELAC PT program criteria, state specific PT program requirements or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside of the Acceptance Limits.
No Evaluation	=	Reported Value cannot be evaluated.

The Method Description is the method the laboratory reported to ERA.

WS Study Discussion:

ERA's WatR™ Supply Proficiency Testing (PT) Study, WS-75, has been reviewed by ERA Senior Management and certified compliant with the requirements of the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document (December 1998), the NELAC Standards Chapter 2 and Associated Appendices, applicable state PT programs, and those contained in the National Institute of Standards and Technology NVLAP Handbooks 150 and 150-19. ERA is a NIST NVLAP accredited PT Provider (Lab Code 2003860).

This Report contains data that are not covered by the NVLAP accreditation.

The opening date of ERA's WatR™ Supply Proficiency Testing Study WS-75 was October 11, 2002, and the closing date was November 25, 2002.

ERA's WatR™ Supply PT Study WS-75 results were examined for any study anomalies. A full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the WS-75 standards met the acceptance criteria contained in the USEPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, and the National Voluntary Laboratory Accreditation Program, Handbook 150-19 for Chemical Calibration for Providers of Proficiency Testing.

The data submitted by participating laboratories were also examined for study anomalies. There were four anomalies found during the review of the study data. These anomalies are addressed on the next page.

The WatR™ Supply PT Study, WS-75, report shall not be reproduced except in its entirety and without the permission of the participating laboratory. The report can not be used by the participating laboratory to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding ERA's WatR™ Supply PT Study, WS-75, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality

NVLAP® Lab code: 200386-0

WS Study Definitions:

WS Study Discussion:

Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

- Study Discussion: WS-75 Unregulated Volatiles - Bromomethane

During the review of the WS-75 Study data ERA discovered a failure rate of 42.1% for bromomethane in the Unregulated Volatiles sample. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'.

Per the requirements of ERA's NIST NVLAP PT Provider Accreditation (Lab code 2003860) and the NELAC Standards (Chapter 2, Section C.4, July 1999), ERA examined all WS-75 data sets for bimodal distribution and/or situations where results from a given method have disproportionally large failure rates. No method biases or multi-modal distributions were found in the WS-75 bromomethane data set. Per the USEPA Criteria Document, the acceptance limits for this compound are ±20% of the assigned value when the concentration is equal to or greater than 10.0 µg/L, as was the case with WS-75, and ±40% of the assigned value when the concentration is below 10.0 µg/L. Of the last twenty-three PT studies conducted by ERA, when bromomethane has been present at a concentration greater than or equal to 10 µg/L, ERA has witnessed an average failure rate of 49.7%. The average failure rate when bromomethane is present at concentrations less than 10 µg/L is 7.64%. The USEPA conducted one study containing bromomethane in the past but a failure rate is not available to ERA for comparison. The acceptance criteria used in that study was 49.9%-139%. Obviously, the USEPA's acceptance criteria are not based on historical data from PT studies. The USEPA criteria require that this analyte be manufactured at concentrations both above and below 10.0 µg/L and randomly assigned in each study. The failure rate for bromomethane in ERA's WS-75 Study would be 7.89% using ERA's statistically determined regression equation. The high failure rate causes us to question the appropriateness of the acceptance limit criteria provided in the USEPA Criteria Document. ERA will communicate this seeming disparity in failure rates to the USEPA. When reviewing each laboratory's performance evaluation for this analyte one must take the above information into consideration.

- Study Discussion: WS-75 Unregulated Volatiles - Fluorotrichloromethane

During the review of the WS-75 Study data ERA discovered a failure rate of 28.6% for fluorotrichloromethane in the Unregulated Volatiles sample. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'.

Per the requirements of ERA's NIST NVLAP PT Provider Accreditation (Lab code 2003860) and the NELAC Standards (Chapter 2, Section C.4, July 1999), ERA examined all WS-75 data sets for bimodal distribution and/or situations where results from a given method have disproportionally large failure rates. No method biases or multi-modal distributions were found in the WS-75 fluorotrichloromethane data set. Per the USEPA Criteria Document, the acceptance limits for this compound are ±20% of the assigned value when the concentration is equal to or greater than 10.0 µg/L, as was the case with WS-75 and ±40% of the assigned value when the concentration is below 10.0 µg/L. Of the last twenty-three PT studies conducted by ERA, when fluorotrichloromethane has been present at a concentration greater than or equal to 10 µg/L, ERA has witnessed an average failure rate of 20.4%. The average failure rate when fluorotrichloromethane is present at concentrations less than 10 µg/L is 2.64%. The USEPA only conducted one study containing fluorotrichloromethane and the failure rate is not available. The USEPA criteria require that this analyte be manufactured at concentrations both above and below 10.0 µg/L and randomly assigned in each study. The failure rate for fluorotrichloromethane in ERA's WS-75 Study would be 20.0% using ERA's statistically determined regression equation. The high failure rate causes us to question the appropriateness of the acceptance limit criteria provided in the USEPA Criteria Document. ERA will communicate this seeming disparity in failure rates to the USEPA. When reviewing each laboratory's performance evaluation for this analyte one must take the above information into consideration.

- Study Discussion: WS-75 Unregulated Volatiles - 1,2,3-Trichloropropane

During the review of the WS-75 Study data ERA discovered a failure rate of 27.8% for 1,2,3-Trichloropropane in the Unregulated Volatiles sample. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'.

Per the requirements of ERA's NIST NVLAP PT Provider Accreditation (Lab code 2003860) and the NELAC Standards (Chapter 2, Section C.4, July 1999), ERA examined all WS-75 data sets for bimodal distribution and/or situations where results from a given method have disproportionally large failure rates. No method biases or multi-modal distributions were found in the WS-75 1,2,3-Trichloropropane data set. Per the USEPA Criteria Document, the acceptance limits for this compound are ±20% of the assigned value when the concentration is equal to or greater than 10.0 µg/L, as was the case with WS-75, and ±40% of the assigned value when the concentration is below 10.0 µg/L. Of the last twenty-three PT studies conducted by ERA, when 1,2,3-Trichloropropane has been present at a concentration greater than or equal to 10 µg/L, ERA has witnessed an average failure rate of 23.9%. The average failure rate when 1,2,3-Trichloropropane is present at concentrations less than 10 µg/L is 8.11%. The USEPA reported a historical failure rate of 17.4% for eight its WS studies. The USEPA criteria require that this analyte be manufactured at concentrations both above and below 10.0 µg/L and randomly assigned in each study. The failure rate for 1,2,3-Trichloropropane in ERA's WS-75 Study would be 11.1% using ERA's statistically determined regression equation. The high failure rate causes us to question the appropriateness of the acceptance limit criteria provided in the USEPA Criteria Document. ERA will communicate this seeming disparity in failure rates to the USEPA. When reviewing each laboratory's performance evaluation for this analyte one must take the above information into consideration.

- Study Discussion: WS-75 Regulated Semivolatiles #1 - Benzo(a)pyrene

During the review of the WS-75 Study data ERA observed a failure rate of 61.1% for benzo(a)pyrene in the Regulated Semivolatiles #1 sample. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification, and stability data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'.

Benzo(a)pyrene was added into the WS-75 Semivolatiles #1 sample at a concentration of 0.237µg/L. The manufacturing range for benzo(a)pyrene is 0.200-2.50µg/L per the EPA's National Standards Criteria Document, December 1998. Eighteen participants submitted results for benzo(a)pyrene. Seven participants submitted "acceptable" results. Of the remaining eleven participants, seven did not detect benzo(a)pyrene and reported "less than" values or 0 µg/L. Four laboratories reported values 2 to 4.5 times the assigned value. The EPA historical mean failure rate was reported as 11.5% for benzo(a)pyrene. ERA's historical mean failure rate for benzo(a)pyrene is 16.6%. ERA has administered 4 studies with the concentration of benzo(a)pyrene less than 1 mg/L. The mean failure rate of these studies is 28.8%, with a range of 0% to 50.0%.

The NELAC Proficiency Testing Reporting Limit (PTRL) for benzo(a)pyrene is 0.070µg/L. The NELAC PTRL was established to provide laboratories with guidance as to the minimum concentration required to analyze for a given analyte. The PTRL is based on the minimum manufacturing concentration required of Proficiency Testing Providers. NELAC has posted this information on it's web site as ERA has also posted this data on our eDATA™2003 web site.

ENVIRONMENTAL
RESOURCE ASSOCIATES®

Study: **WS75**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Laboratories**

Report Type: **Complete**

Report Method: **Method A**

5540 Marshall St., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-75 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Laboratories
Suite B
15130 S Keeler
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/17/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Metals							
1000	Aluminum	µg/L	452	476	423 - 529	Acceptable	EPA 200.8
0140	Antimony †	µg/L	19.7	18.9	13.2 - 24.6	Acceptable	EPA 200.8
0001	Arsenic †	µg/L	58.3	60.4	52.8 - 67.5	Acceptable	EPA 200.8
0002	Barium †	µg/L	883	904	768 - 1040	Acceptable	EPA 200.8
0141	Beryllium †	µg/L	8.77	8.84	7.51 - 10.2	Acceptable	EPA 200.8
0226	Boron †	µg/L	957	985	916 - 1090	Acceptable	EPA 200.8
0003	Cadmium †	µg/L	7.57	8.00	6.40 - 9.60	Acceptable	EPA 200.8
0004	Chromium †	µg/L	83.3	81.9	69.6 - 94.2	Acceptable	EPA 200.8
0091	Copper †	µg/L	414	387	348 - 426	Acceptable	EPA 200.8
1070	Iron	µg/L	501	512	469 - 557	Acceptable	EPA 200.8
0005	Lead †	µg/L	32.7	32.9	23.0 - 42.8	Acceptable	EPA 200.8
0236	Manganese †	µg/L	731	691	644 - 726	Not Acceptable	EPA 200.8
0237	Molybdenum †	µg/L	13.7	13.4	10.8 - 16.1	Acceptable	EPA 200.8
0142	Nickel †	µg/L	169	162	138 - 186	Acceptable	EPA 200.8
0007	Selenium †	µg/L	30.9	30.0	24.0 - 36.0	Acceptable	EPA 200.8
1150	Silver	µg/L	112	109	98.3 - 120	Acceptable	EPA 200.8
0143	Thallium †	µg/L	5.38	5.47	3.83 - 7.11	Acceptable	EPA 200.8
1185	Vanadium	µg/L	417	388	361 - 412	Not Acceptable	EPA 200.7
0239	Zinc †	µg/L	851	806	740 - 867	Acceptable	EPA 200.8
Mercury							
0006	Mercury †	µg/L	4.09	3.95	2.77 - 5.14	Acceptable	EPA 200.8

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-75 Final Complete Report

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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/17/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Volatiles							
0039	Benzene †	µg/L	17.4	16.4	13.1 - 19.7	Acceptable	EPA 524.2
0037	Carbon tetrachloride †	µg/L	3.46	3.19	1.91 - 4.47	Acceptable	EPA 524.2
0049	Chlorobenzene †	µg/L	5.26	4.67	2.80 - 6.54	Acceptable	EPA 524.2
0054	1,2-Dichlorobenzene †	µg/L	11.2	10.0	8.00 - 12.0	Acceptable	EPA 524.2
0041	1,4-Dichlorobenzene †	µg/L	13.1	11.6	9.28 - 13.9	Acceptable	EPA 524.2
0035	1,2-Dichloroethane †	µg/L	10.4	9.47	5.68 - 13.3	Acceptable	EPA 524.2
0034	1,1-Dichloroethylene †	µg/L	12.1	11.3	9.04 - 13.6	Acceptable	EPA 524.2
0043	cis-1,2-Dichloroethylene †	µg/L	38.8	37.2	29.8 - 44.6	Acceptable	EPA 524.2
0042	trans-1,2-Dichloroethylene †	µg/L	10.1	9.82	5.89 - 13.7	Acceptable	EPA 524.2
0044	1,2-Dichloropropane †	µg/L	8.61	8.15	4.89 - 11.4	Acceptable	EPA 524.2
0048	Ethylbenzene †	µg/L	3.93	3.35	2.01 - 4.69	Acceptable	EPA 524.2
0055	Methylene chloride †	µg/L	11.6	11.1	8.88 - 13.3	Acceptable	EPA 524.2
0053	Styrene †	µg/L	14.4	12.6	10.1 - 15.1	Acceptable	EPA 524.2
0040	Tetrachloroethylene †	µg/L	17.3	16.0	12.8 - 19.2	Acceptable	EPA 524.2
0047	Toluene †	µg/L	15.7	14.6	11.7 - 17.5	Acceptable	EPA 524.2
0076	1,2,4-Trichlorobenzene †	µg/L	10.1	13.8	11.0 - 16.6	Not Acceptable	EPA 524.2
0036	1,1,1-Trichloroethane †	µg/L	5.98	5.54	3.32 - 7.76	Acceptable	EPA 524.2
0061	1,1,2-Trichloroethane †	µg/L	10.3	9.44	5.66 - 13.2	Acceptable	EPA 524.2
0038	Trichloroethylene †	µg/L	17.6	16.8	13.4 - 20.2	Acceptable	EPA 524.2
0032	Vinyl chloride †	µg/L	11.3	10.0	6.00 - 14.0	Acceptable	EPA 524.2
0090	Xylenes, total †	µg/L	36.4	29.5	23.6 - 35.4	Not Acceptable	EPA 524.2
Halomethanes							
0019	Bromodichloromethane †	µg/L	23.2	12.9	10.3 - 15.5	Not Acceptable	EPA 524.2
0018	Bromoform †	µg/L	7.89	7.15	4.29 - 10.0	Acceptable	EPA 524.2
0020	Chlorodibromomethane †	µg/L	17.3	15.6	12.5 - 18.7	Acceptable	EPA 524.2
0017	Chloroform †	µg/L	23.2	20.9	16.7 - 25.1	Acceptable	EPA 524.2
0021	Total Trihalomethanes †	µg/L	62.7	56.6	45.3 - 67.9	Acceptable	EPA 524.2
Unregulated Volatiles							
0067	Bromobenzene †	µg/L	7.18	6.62	3.97 - 9.27	Acceptable	EPA 524.2
0089	Bromochloromethane †	µg/L	5.90	5.86	3.52 - 8.20	Acceptable	EPA 524.2
0069	Bromomethane †	µg/L	7.70	10.0	8.00 - 12.0	Not Acceptable	EPA 524.2
0079	n-Butylbenzene †	µg/L	14.3	13.5	10.8 - 16.2	Acceptable	EPA 524.2
0086	sec-Butylbenzene †	µg/L	3.90	3.54	2.12 - 4.96	Acceptable	EPA 524.2
0085	tert-Butylbenzene †	µg/L	14.7	14.2	11.4 - 17.0	Acceptable	EPA 524.2
5000	tert-Butylmethyl ether (MTBE)	µg/L	< 0.5	0.00		Acceptable	EPA 524.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

Tenkasi S. Viswanathan
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Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/17/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Unregulated Volatiles (Cont.)							
0070	Chloroethane †	µg/L	< 1.2	0.00		Acceptable	EPA 524.2
0068	Chloromethane †	µg/L	< 3.0	0.00		Acceptable	EPA 524.2
0071	2-Chlorotoluene †	µg/L	< 1.2	0.00		Acceptable	EPA 524.2
0072	4-Chlorotoluene †	µg/L	9.02	8.41	5.05 - 11.8	Acceptable	EPA 524.2
0057	Dibromomethane †	µg/L	< 1.2	0.00		Acceptable	EPA 524.2
0066	1,3-Dichlorobenzene †	µg/L	8.38	7.26	4.36 - 10.2	Acceptable	EPA 524.2
0088	Dichlorodifluoromethane †	µg/L	< 1.2	0.00		Acceptable	EPA 524.2
0056	1,1-Dichloroethane †	µg/L	6.27	5.72	3.43 - 8.01	Acceptable	EPA 524.2
0059	1,3-Dichloropropane †	µg/L	11.4	12.8	10.2 - 15.4	Acceptable	EPA 524.2
0060	2,2-Dichloropropane †	µg/L	< 3.0	0.00		Acceptable	EPA 524.2
0058	1,1-Dichloropropene †	µg/L	< 1.2	0.00		Acceptable	EPA 524.2
0152	cis-1,3-Dichloropropene †	µg/L	4.07	4.30	2.58 - 6.02	Acceptable	EPA 524.2
0153	trans-1,3-Dichloropropene †	µg/L	5.65	5.90	3.54 - 8.26	Acceptable	EPA 524.2
0087	Fluorotrichloromethane †	µg/L	19.5	18.8	15.0 - 22.6	Acceptable	EPA 524.2
0081	Hexachlorobutadiene †	µg/L	< 1.5	0.00		Acceptable	EPA 524.2
0084	Isopropylbenzene †	µg/L	7.49	7.82	4.69 - 10.9	Acceptable	EPA 524.2
0083	4-Isopropyltoluene †	µg/L	12.1	10.6	8.48 - 12.7	Acceptable	EPA 524.2
0078	n-Propylbenzene †	µg/L	16.9	15.5	12.4 - 18.6	Acceptable	EPA 524.2
0063	1,1,1,2-Tetrachloroethane †	µg/L	8.65	9.12	5.47 - 12.8	Acceptable	EPA 524.2
0065	1,1,2,2-Tetrachloroethane †	µg/L	8.93	8.30	4.98 - 11.6	Acceptable	EPA 524.2
0077	1,2,3-Trichlorobenzene †	µg/L	5.37	4.77	2.86 - 6.68	Acceptable	EPA 524.2
0064	1,2,3-Trichloropropane †	µg/L	18.4	17.2	13.8 - 20.6	Acceptable	EPA 524.2
0075	1,2,4-Trimethylbenzene †	µg/L	5.75	5.22	3.13 - 7.31	Acceptable	EPA 524.2
0082	1,3,5-Trimethylbenzene †	µg/L	12.4	11.1	8.88 - 13.3	Acceptable	EPA 524.2
Regulated Pesticides							
0093	Alachlor †	µg/L	18.2	17.0	9.35 - 24.7	Acceptable	EPA 525.2
0256	Aldrin †	µg/L		1.49	0.630 - 1.87		
0094	Atrazine †	µg/L	21.9	13.9	7.65 - 20.2	Not Acceptable	EPA 525.2
7160	Butachlor	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
7410	Diazinon	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
0258	Dieldrin †	µg/L		1.70	1.19 - 2.13		
0011	Endrin †	µg/L		4.74	3.32 - 6.16		
0095	Heptachlor †	µg/L		4.02	2.21 - 5.83		
0096	Heptachlor epoxide (beta) †	µg/L		0.292	0.161 - 0.423		
0172	Hexachlorobenzene †	µg/L	1.7	1.19	0.540 - 1.55	Not Acceptable	EPA 525.2
0112	Hexachlorocyclopentadiene †	µg/L	2.5	4.37	0.114 - 5.58	Acceptable	EPA 525.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/17/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Pesticides (Cont.)							
0012	gamma-BHC (Lindane) †	µg/L		0.469	0.258 - 0.680		
0013	Methoxychlor †	µg/L		15.7	8.64 - 22.8		
7835	Metolachlor	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
7845	Metribuzin	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
7875	Molinate (ordram)	µg/L	15.1	9.96	5.48 - 14.4	Not Acceptable	EPA 525.2
8035	Prometon	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
0259	Propachlor †	µg/L	2.16	1.60	0.888 - 2.36	Acceptable	EPA 525.2
0113	Simazine †	µg/L	9.29	10.1	2.38 - 15.3	Acceptable	EPA 525.2
8220	Thiobencarb	µg/L		0.00			
0244	Trifluralin †	µg/L	1.6	1.21	0.424 - 1.39	Not Acceptable	EPA 525.2
EDB/DBCP							
0045	Dibromochloropropane (DBCP) †	µg/L	0.725	0.664	0.398 - 0.930	Acceptable	EPA 504.1
0046	Ethylene Dibromide (EDB) †	µg/L	1.03	0.934	0.560 - 1.31	Acceptable	EPA 504.1
5180	1,2,3-Trichloropropane	µg/L	1.99	1.84	1.10 - 2.58	Acceptable	EPA 504.1
Regulated Semivolatiles #1							
5500	Acenaphthene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5505	Acenaphthylene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5555	Anthracene	µg/L	11.3	11.4	5.70 - 17.1	Acceptable	EPA 525.2
5575	Benzo(a)anthracene	µg/L	24.8	26.6	13.3 - 39.9	Acceptable	EPA 525.2
5585	Benzo(b)fluoranthene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5600	Benzo(k)fluoranthene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5590	Benzo(g,h,i)perylene	µg/L	3.70	5.53	2.77 - 8.30	Acceptable	EPA 525.2
0122	Benzo(a)pyrene †	µg/L	< 0.07	0.237	0.0815 - 0.346	Not Acceptable	EPA 525.2
5670	Butylbenzylphthalate	µg/L	< 0.5	0.00		Acceptable	EPA 525.2
5855	Chrysene	µg/L	25.1	28.6	14.3 - 42.9	Acceptable	EPA 525.2
5895	Dibenz(a,h)anthracene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5925	Di-n-butylphthalate	µg/L	< 2.0	0.00		Acceptable	EPA 525.2
6070	Diethylphthalate	µg/L	< 0.5	0.00		Acceptable	EPA 525.2
6135	Dimethylphthalate	µg/L	6.43	5.19	1.56 - 8.82	Acceptable	EPA 525.2
6200	Di-n-octylphthalate	µg/L	28.0	40.0	3.36 - 70.4	Acceptable	EPA 525.2
0134	bis(2-Ethylhexyl)adipate †	µg/L	16.4	19.9	7.20 - 29.1	Acceptable	EPA 525.2
0136	bis(2-Ethylhexyl)phthalate †	µg/L	14.9	21.2	8.73 - 32.9	Acceptable	EPA 525.2
6265	Fluoranthene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
6270	Fluorene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
6315	Indeno(1,2,3-cd)pyrene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/17/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Semivolatiles #1 (Cont.)							
6380	1-Methylnaphthalene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
6385	2-Methylnaphthalene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5005	Naphthalene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
6615	Phenanthrene	µg/L	9.39	8.13	4.07 - 12.2	Acceptable	EPA 525.2
6665	Pyrene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-75 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Laboratories
Suite B
15130 S Keeler
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/17/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Perchlorate							
1895	Perchlorate	µg/L	15.9	16.1	12.1 - 20.1	Acceptable	EPA 314

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

Study: **WS75**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Laboratories**

Report Type: **Complete**

Report Method: **Method B**

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-75 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Laboratories
Suite B
15130 S Keeler
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/17/02

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Metals							
1000	Aluminum	µg/L		476	423 - 529		
0140	Antimony †	µg/L		18.9	13.2 - 24.6		
0001	Arsenic †	µg/L		60.4	52.8 - 67.5		
0002	Barium †	µg/L		904	768 - 1040		
0141	Beryllium †	µg/L		8.84	7.51 - 10.2		
0226	Boron †	µg/L		985	916 - 1090		
0003	Cadmium †	µg/L		8.00	6.40 - 9.60		
0004	Chromium †	µg/L		81.9	69.6 - 94.2		
0091	Copper †	µg/L		387	348 - 426		
1070	Iron	µg/L		512	469 - 557		
0005	Lead †	µg/L		32.9	23.0 - 42.8		
0236	Manganese †	µg/L		691	644 - 726		
0237	Molybdenum †	µg/L		13.4	10.8 - 16.1		
0142	Nickel †	µg/L		162	138 - 186		
0007	Selenium †	µg/L		30.0	24.0 - 36.0		
1150	Silver	µg/L	112	109	98.3 - 120	Acceptable	EPA 200.7
0143	Thallium †	µg/L		5.47	3.83 - 7.11		
1185	Vanadium	µg/L		388	361 - 412		
0239	Zinc †	µg/L		806	740 - 867		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

WS-81 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 **EPA ID: KS00902** **Report Issued: 06/19/03**

WS Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Values are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, and the National Environmental Laboratory Accreditation Conference (NELAC) program criteria as applicable. A parameter not added to the standard is given an Assigned Value of "0" per the guidelines contained in the USEPA's Criteria Document and NELAC standards.

The Acceptance Limits are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, the NELAC PT program criteria or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside of the Acceptance Limits.
No Evaluation	=	Reported Value cannot be evaluated.

The Method Description is the method the laboratory reported to ERA.

WS Study Discussion:

ERA's WatR™Supply Proficiency Testing Study, WS-81, has been reviewed by ERA Senior Management and certified compliant with the requirements of the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document (December 1998), the NELAC Standards Chapter 2 and Associated Appendices, applicable state PT programs, and those contained in the National Institute of Standards and Technology NVLAP Handbooks 150 and 150-19. ERA is a NIST NVLAP accredited PT Provider (Lab Code 2003860).

This report contains data that are not covered by the NVLAP accreditation.

The opening date of ERA's WatR™Supply Study, WS-81, was April 14, 2003, and the closing date was May 29, 2003.

ERA's WatR™Supply Study, WS-81, standards were examined for any anomalies. A full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the WS-81 standards met the acceptance criteria contained in the USEPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, and the National Voluntary Laboratory Accreditation Program Handbook 150-19 for Chemical Calibration for Providers of Proficiency Testing.

The data submitted by participating laboratories was also examined for study anomalies. There were two anomalies found during the review of the study data. These anomalies are addressed on the next page.

WatR™Supply Study, WS-81, reports shall not be reproduced except in their entirety and not without the permission of the participating laboratories. The report must not be used by the participating laboratories to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding ERA's WatR™Supply Proficiency Testing Study, WS-81, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood,

NVLAP® Lab code: 200386-0

WS Study Definitions:

WS Study Discussion:

Quality Assurance Director, at 1-800-372-0122.

- Study Discussion: WS-81 Unregulated Volatiles - 1,2,3-Trichlorobenzene

During the review of the WS-81 Study data ERA discovered a failure rate of 21.4% for 1,2,3-Trichlorobenzene in the Unregulated Volatiles sample. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'.

Per the requirements of ERA's NIST NVLAP PT Provider Accreditation (Lab code 2003860) and the NELAC Standards (Chapter 2, Section C.4, July 1999), ERA examined all WS-81 data sets for bimodal distribution and/or situations where results from a given method have disproportionately large failure rates. No method biases or multi-modal distributions were found in the WS-81 1,2,3-Trichlorobenzene data set. Per the USEPA Criteria Document, the acceptance limits for this compound are ±20% of the assigned value when the concentration is equal to or greater than 10.0µg/L, as was the case with WS-81, and ±40% of the assigned value when the concentration is below 10.0µg/L. Of the last twenty-seven PT studies conducted by ERA, when 1,2,3-Trichlorobenzene has been present at a concentration greater than or equal to 10µg/L, ERA has witnessed an average failure rate of 19.6%. The average failure rate when 1,2,3-Trichlorobenzene has been present at concentrations less than 10µg/L is 5.31%. The USEPA's historical failure rate for 1,2,3-Trichlorobenzene is 14.4%. The USEPA criteria require that this analyte be manufactured at concentrations both above and below 10.0µg/L and randomly assigned in each study. The failure rate for 1,2,3-Trichlorobenzene in ERA's WS-81 Study would be 11.9% using ERA's statistically determined regression equation. The high failure rate causes us to question the appropriateness of the acceptance limit criteria provided in the USEPA Criteria Document. ERA will communicate this seeming disparity in failure rates to NIST/NVLAP. When reviewing each laboratory's performance evaluation for this analyte one must take the above information into consideration.

- Study Discussion: WS-81 Unregulated Volatiles - Hexachlorobutadiene

During the review of the WS-81 Study data ERA discovered a failure rate of 22.0% for hexachlorobutadiene in the Unregulated Volatiles sample. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'.

Per the requirements of ERA's NIST NVLAP PT Provider Accreditation (Lab code 2003860) and the NELAC Standards (Chapter 2, Section C.4, July 1999), ERA examined all WS-81 data sets for bimodal distribution and/or situations where results from a given method have disproportionately large failure rates. No method biases or multi-modal distributions were found in the WS-81 hexachlorobutadiene data set. Per the USEPA Criteria Document, the acceptance limits for this compound are ±20% of the assigned value when the concentration is equal to or greater than 10.0µg/L, as was the case with WS-81, and ±40% of the assigned value when the concentration is below 10.0µg/L. Of the last twenty-two PT studies conducted by ERA, when hexachlorobutadiene has been present at a concentration greater than or equal to 10µg/L, ERA has witnessed an average failure rate of 15.4%. The average failure rate when hexachlorobutadiene has been present at concentrations less than 10µg/L is 3.24%. The USEPA's historical failure rate for hexachlorobutadiene is 14.5%. The USEPA criteria require that this analyte be manufactured at concentrations both above and below 10.0µg/L and randomly assigned in each study. The failure rate for hexachlorobutadiene in ERA's WS-81 Study would be 14.6% using ERA's statistically determined regression equation. The high failure rate causes us to question the appropriateness of the acceptance limit criteria provided in the USEPA Criteria Document. ERA will communicate this seeming disparity in failure rates to NIST/NVLAP. When reviewing each laboratory's performance evaluation for this analyte one must take the above information into consideration.

ENVIRONMENTAL
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Study: **WS-81**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method A**

ACCREDITED
Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP
Lab Code: 200386-0

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-81 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
Suite B
15130 S Keeler
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/19/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Metals							
1000	Aluminum	µg/L	923	893	803 - 980	Acceptable	EPA 200.7
0140	Antimony †	µg/L	17.7	19.2	13.4 - 25.0	Acceptable	EPA 200.8
0001	Arsenic †	µg/L	76.1	82.6	72.3 - 92.2	Acceptable	EPA 200.8
0002	Barium †	µg/L	773	773	657 - 889	Acceptable	EPA 200.7
0141	Beryllium †	µg/L	3.67	3.31	2.81 - 3.81	Acceptable	EPA 200.7
0226	Boron †	µg/L	895	891	829 - 983	Acceptable	EPA 200.8
0003	Cadmium †	µg/L	18.2	16.7	13.4 - 20.0	Acceptable	EPA 200.7
0004	Chromium †	µg/L	190	180	153 - 207	Acceptable	EPA 200.7
0091	Copper †	µg/L	111	106	95.4 - 117	Acceptable	EPA 200.7
1070	Iron	µg/L	166	157	142 - 174	Acceptable	EPA 200.7
0005	Lead †	µg/L	25.3	27.0	18.9 - 35.1	Acceptable	EPA 200.8
0236	Manganese †	µg/L	335	320	297 - 337	Acceptable	EPA 200.7
0237	Molybdenum †	µg/L	37.7	37.3	31.1 - 43.4	Acceptable	EPA 200.7
0142	Nickel †	µg/L	299	276	235 - 317	Acceptable	EPA 200.7
0007	Selenium †	µg/L	37.6	41.7	33.4 - 50.0	Acceptable	EPA 200.8
1150	Silver	µg/L	54.3	48.2	43.2 - 53.0	Not Acceptable	EPA 200.7
0143	Thallium †	µg/L	3.81	4.25	2.98 - 5.53	Acceptable	EPA 200.8
1185	Vanadium	µg/L	292	288	267 - 306	Acceptable	EPA 200.7
0239	Zinc †	µg/L	778	707	649 - 761	Not Acceptable	EPA 200.7
Mercury							
0006	Mercury †	µg/L	7.58	8.47	5.93 - 11.0	Acceptable	EPA 200.8
Inorganics							
1575	Chloride	mg/L	53.2	56.7	52.6 - 60.3	Acceptable	EPA 300.0
1610	Conductivity	µmhos/cm	563	454	430 - 478	Not Acceptable	EPA 120.1
0010	Fluoride †	mg/L	2.86	3.18	2.86 - 3.50	Acceptable	EPA 300.0
0009	Nitrate as N †	mg/L	6.42	6.99	6.29 - 7.69	Acceptable	EPA 300.0
1820	Nitrate + Nitrite as N	mg/L	6.42	6.99	6.29 - 7.69	Acceptable	EPA 300.0
1125	Potassium	mg/L	25.4	26.1	23.7 - 28.6	Acceptable	EPA 200.7
0145	Sulfate †	mg/L	23.4	23.7	20.8 - 26.3	Acceptable	EPA 300.0
0024	Total Dissolved Solids at 180°C †	mg/L	348	340	220 - 460	Acceptable	EPA 160.1
TOC							
0263	Total organic carbon †	mg/L	3.38	2.40	2.09 - 2.86	Not Acceptable	EPA 415.1

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-81 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
Suite B
15130 S Keeler
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/19/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Volatiles							
0039	Benzene †	µg/L	9.91	10.7	8.56 - 12.8	Acceptable	524.2
0037	Carbon tetrachloride †	µg/L	17.4	18.2	14.6 - 21.8	Acceptable	524.2
0049	Chlorobenzene †	µg/L	13.7	14.5	11.6 - 17.4	Acceptable	524.2
0054	1,2-Dichlorobenzene †	µg/L	16.4	16.4	13.1 - 19.7	Acceptable	524.2
0041	1,4-Dichlorobenzene †	µg/L	8.98	9.08	5.45 - 12.7	Acceptable	524.2
0035	1,2-Dichloroethane †	µg/L	6.99	7.18	4.31 - 10.1	Acceptable	524.2
0034	1,1-Dichloroethylene †	µg/L	17.8	18.5	14.8 - 22.2	Acceptable	524.2
0043	cis-1,2-Dichloroethylene †	µg/L	11.6	12.2	9.76 - 14.6	Acceptable	524.2
0042	trans-1,2-Dichloroethylene †	µg/L	12.9	14.0	11.2 - 16.8	Acceptable	524.2
0044	1,2-Dichloropropane †	µg/L	9.70	9.94	5.96 - 13.9	Acceptable	524.2
0048	Ethylbenzene †	µg/L	3.43	3.81	2.29 - 5.33	Acceptable	524.2
0055	Methylene chloride †	µg/L	11.4	13.5	10.8 - 16.2	Acceptable	524.2
0053	Styrene †	µg/L	13.8	14.4	11.5 - 17.3	Acceptable	524.2
0040	Tetrachloroethylene †	µg/L	16.6	17.6	14.1 - 21.1	Acceptable	524.2
0047	Toluene †	µg/L	16.1	17.1	13.7 - 20.5	Acceptable	524.4
0076	1,2,4-Trichlorobenzene †	µg/L	7.38	3.43	2.06 - 4.80	Not Acceptable	524.2
0036	1,1,1-Trichloroethane †	µg/L	10.8	11.7	9.36 - 14.0	Acceptable	524.2
0061	1,1,2-Trichloroethane †	µg/L	17.6	18.3	14.6 - 22.0	Acceptable	524.2
0038	Trichloroethylene †	µg/L	9.22	9.87	5.92 - 13.8	Acceptable	524.2
0032	Vinyl chloride †	µg/L	3.82	4.29	2.57 - 6.01	Acceptable	524.2
0090	Xylenes, total †	µg/L	26.1	27.9	22.3 - 33.5	Acceptable	524.2
Halomethanes							
0019	Bromodichloromethane †	µg/L	39.4	38.7	31.0 - 46.4	Acceptable	524.2
0018	Bromoform †	µg/L	12.1	13.7	11.0 - 16.4	Acceptable	524.2
0020	Chlorodibromomethane †	µg/L	26.4	27.6	22.1 - 33.1	Acceptable	524.2
0017	Chloroform †	µg/L	6.46	6.97	4.18 - 9.76	Acceptable	524.2
0021	Total Trihalomethanes †	µg/L	84.4	87.0	69.6 - 104	Acceptable	524.2
Unregulated Volatiles							
0067	Bromobenzene †	µg/L	9.48	9.46	5.68 - 13.2	Acceptable	EPA 524.2
0089	Bromochloromethane †	µg/L	3.89	4.19	2.51 - 5.87	Acceptable	EPA 524.2
0069	Bromomethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0079	n-Butylbenzene †	µg/L	6.74	6.98	4.19 - 9.77	Acceptable	EPA 524.2
0086	sec-Butylbenzene †	µg/L	3.34	0.00		Not Acceptable	EPA 524.2
0085	tert-Butylbenzene †	µg/L	4.51	4.84	2.90 - 6.78	Acceptable	EPA 524.2
5000	tert-Butylmethyl ether (MTBE)	µg/L	< 0.5	0.00		Acceptable	EPA 524.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-81 Final Complete Report

Tenkasi S. Viswanathan
QA Director
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Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/19/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Unregulated Volatiles (Cont.)							
0070	Chloroethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0068	Chloromethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0071	2-Chlorotoluene †	µg/L	5.32	5.00	3.00 - 7.00	Acceptable	EPA 524.2
0072	4-Chlorotoluene †	µg/L	15.3	14.5	11.6 - 17.4	Acceptable	EPA 524.2
0057	Dibromomethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0066	1,3-Dichlorobenzene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0088	Dichlorodifluoromethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0056	1,1-Dichloroethane †	µg/L	5.15	5.80	3.48 - 8.12	Acceptable	EPA 524.2
0059	1,3-Dichloropropane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0060	2,2-Dichloropropane †	µg/L	5.38	6.29	3.77 - 8.81	Acceptable	EPA 524.2
0058	1,1-Dichloropropene †	µg/L	17.4	18.1	14.5 - 21.7	Acceptable	EPA 524.2
0152	cis-1,3-Dichloropropene †	µg/L	5.29	6.17	3.70 - 8.64	Acceptable	EPA 524.2
0153	trans-1,3-Dichloropropene †	µg/L	9.92	10.9	8.72 - 13.1	Acceptable	EPA 524.2
0087	Fluorotrichloromethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0081	Hexachlorobutadiene †	µg/L	14.0	13.1	10.5 - 15.7	Acceptable	EPA 524.2
0084	Isopropylbenzene †	µg/L	4.32	4.75	2.85 - 6.65	Acceptable	EPA 524.2
0083	4-Isopropyltoluene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0078	n-Propylbenzene †	µg/L	3.65	3.77	2.26 - 5.28	Acceptable	EPA 524.2
0063	1,1,1,2-Tetrachloroethane †	µg/L	10.1	11.1	8.88 - 13.3	Acceptable	EPA 524.2
0065	1,1,2,2-Tetrachloroethane †	µg/L	3.26	3.46	2.08 - 4.84	Acceptable	EPA 524.2
0077	1,2,3-Trichlorobenzene †	µg/L	20.1	14.3	11.4 - 17.2	Not Acceptable	EPA 524.2
0064	1,2,3-Trichloropropane †	µg/L	4.87	4.89	2.93 - 6.85	Acceptable	EPA 524.2
0075	1,2,4-Trimethylbenzene †	µg/L	10.8	10.6	8.48 - 12.7	Acceptable	EPA 524.2
0082	1,3,5-Trimethylbenzene †	µg/L	6.49	6.65	3.99 - 9.31	Acceptable	EPA 524.2
Regulated Pesticides							
0093	Alachlor †	µg/L	15.7	17.9	9.85 - 26.0	Acceptable	525.2
0256	Aldrin †	µg/L	1.1	1.30	0.548 - 1.63	Acceptable	508.1
0094	Atrazine †	µg/L	4.55	7.43	4.09 - 10.8	Acceptable	525.2
7160	Butachlor	µg/L		0.00			
7410	Diazinon	µg/L		0.00			
0258	Dieldrin †	µg/L	1.80	2.02	1.41 - 2.53	Acceptable	508.1
0011	Endrin †	µg/L	4.74	4.04	2.83 - 5.25	Acceptable	508.1
0095	Heptachlor †	µg/L	3.58	3.77	2.07 - 5.47	Acceptable	508.1
0096	Heptachlor epoxide (beta) †	µg/L	2.55	2.44	1.34 - 3.54	Acceptable	508.1
0172	Hexachlorobenzene †	µg/L	1.99	2.95	1.36 - 3.74	Acceptable	525.2
0112	Hexachlorocyclopentadiene †	µg/L	1.44	2.82	0.123 - 3.60	Acceptable	525.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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WS-81 Final Complete Report

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QA Director
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15130 S Keeler
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/19/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Pesticides (Cont.)							
0012	gamma-BHC (Lindane) †	µg/L	0.697	1.10	0.605 - 1.60	Acceptable	508.1
0013	Methoxychlor †	µg/L	10.6	12.1	6.66 - 17.5	Acceptable	508.1
7835	Metolachlor	µg/L		19.5	8.81 - 29.1		
7845	Metribuzin	µg/L		0.00			
7875	Molinate (ordram)	µg/L		8.30	3.93 - 11.9		
8035	Prometon	µg/L		0.00			
0259	Propachlor †	µg/L		1.73	0.976 - 2.52		
0113	Simazine †	µg/L	4.52	13.5	3.11 - 19.9	Acceptable	525.2
8220	Thiobencarb	µg/L		0.00			
0244	Trifluralin †	µg/L		1.13	0.384 - 1.27		
Toxaphene							
0014	Toxaphene †	µg/L	13.8	15.8	8.69 - 22.9	Acceptable	EPA 508.1
Chlordane							
0097	Chlordane, technical †	µg/L	13.7	18.9	10.4 - 27.4	Acceptable	EPA 508.1
EDB/DBCP							
0045	Dibromochloropropane (DBCP) †	µg/L	1.20	1.21	0.726 - 1.69	Acceptable	EPA 504.1
0046	Ethylene Dibromide (EDB) †	µg/L	0.647	0.705	0.423 - 0.987	Acceptable	EPA 504.1
5180	1,2,3-Trichloropropane	µg/L	1.64	1.68	0.847 - 2.34	Acceptable	EPA 504.1
Regulated Semivolatiles #1							
5500	Acenaphthene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5505	Acenaphthylene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5555	Anthracene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5575	Benzo(a)anthracene	µg/L	30.5	40.8	17.0 - 49.4	Acceptable	EPA 525.2
5585	Benzo(b)fluoranthene	µg/L	2.42	2.39	1.11 - 3.27	Acceptable	EPA 525.2
5600	Benzo(k)fluoranthene	µg/L	2.44	2.38	1.11 - 3.33	Acceptable	EPA 525.2
5590	Benzo(g,h,i)perylene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
0122	Benzo(a)pyrene †	µg/L	1.04	1.14	0.280 - 1.48	Acceptable	EPA 525.2
5670	Butylbenzylphthalate	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5855	Chrysene	µg/L	18.2	17.4	9.03 - 23.3	Acceptable	EPA 525.2
5895	Dibenz(a,h)anthracene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5925	Di-n-butylphthalate	µg/L	< 0.5	0.00		Acceptable	EPA 525.2
6070	Diethylphthalate	µg/L	33.9	39.3	12.2 - 58.2	Acceptable	EPA 525.2
6135	Dimethylphthalate	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
6200	Di-n-octylphthalate	µg/L	< 0.2	0.00		Acceptable	EPA 525.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-81 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
Suite B
15130 S Keeler
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/19/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Semivolatiles #1 (Cont.)							
0134	bis(2-Ethylhexyl)adipate †	µg/L	17.7	23.8	9.03 - 34.7	Acceptable	EPA 525.2
0136	bis(2-Ethylhexyl)phthalate †	µg/L	36.6	43.8	19.0 - 68.3	Acceptable	EPA 525.2
6265	Fluoranthene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
6270	Fluorene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
6315	Indeno(1,2,3-cd)pyrene	µg/L	11.0	12.6	4.74 - 16.1	Acceptable	EPA 525.2
5005	Naphthalene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
6615	Phenanthrene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
6665	Pyrene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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Regulatory Agencies	Agency Requested Report Type	Agency Lab ID	Contact
Alabama	Complete Report		John Gable
Florida	Complete Report		Steve Arms
Georgia	Complete Report		Loretta M. Lambert
Kansas	Complete Report	E-10254	MAREN FARR
North Carolina (WS)	Complete Report		Mike King
South Carolina	Complete Report		Connie Turner
Tennessee	Complete Report		Charles Mickle

WS-87 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 **EPA ID: KS00902**

Report Issued: 12/15/03
Study Dates: 10/10/03 - 11/24/03

WS Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Values are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, and the National Environmental Laboratory Accreditation Conference (NELAC) program criteria as applicable. A parameter not added to the standard is given an Assigned Value of "0" per the guidelines contained in the USEPA's Criteria Document and NELAC standards.

The Acceptance Limits are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, the NELAC PT program criteria or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside of the Acceptance Limits.
No Evaluation	=	Reported Value cannot be evaluated.

The Method Description is the method the laboratory reported to ERA.

WS Study Discussion:

ERA's WatR™ Supply Proficiency Testing Study, WS-87, has been reviewed by ERA Senior Management and certified compliant with the requirements of the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document (December 1998), the NELAC Standards Chapter 2 and Associated Appendices, applicable state PT programs, and those contained in the National Institute of Standards and Technology NVLAP Handbooks 150 and 150-19. ERA is a NIST NVLAP accredited PT Provider (Lab Code 2003860).

This report contains data that are not covered by the NVLAP accreditation.

ERA's WatR™ Supply Study, WS-87, standards were examined for any anomalies. A full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the WS-87 standards met the acceptance criteria contained in the USEPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, and the National Voluntary Laboratory Accreditation Program Handbook 150-19 for Chemical Calibration for Providers of Proficiency Testing.

The data submitted by participating laboratories was also examined for study anomalies. There were three anomalies found during the review of the study data. These anomalies are addressed on the following page.

WatR™ Supply Study, WS-87, reports shall not be reproduced except in their entirety and not without the permission of the participating laboratories. The report must not be used by the participating laboratories to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding ERA's WatR™ Supply Proficiency Testing Study, WS-87, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

- Study Discussion: WS-87 Unregulated Volatiles - Chloromethane

During the review of the WS-87 Study data ERA discovered a failure rate of 63.3% for chloromethane in the Unregulated Volatiles sample. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'.

Per the requirements of ERA's NIST NVLAP PT Provider Accreditation (Lab code 2003860) and the NELAC Standards (July 2003), ERA examined all WS-87 data sets for bimodal distribution and/or situations where results from a given method have disproportionally large failure rates. No method biases or multi-modal distributions were found in the WS-87 chloromethane data set. Per the USEPA Criteria Document, the acceptance limits for this compound are ±20% of the assigned value when the concentration is equal to or greater than 10.0 µg/L, as was the case with WS-87, and ±40% of the assigned value when the concentration is below 10.0 µg/L. Of the last thirty-five PT studies conducted by ERA, when chloromethane has been present at a concentration greater than or equal to 10 µg/L, ERA has witnessed an average failure rate of 40.6%. The average failure rate when chloromethane has been present at concentrations less than 10 µg/L is 14.1%. To the best of our knowledge the USEPA has never conducted a study containing chloromethane so a failure rate is not available to ERA for comparison. Obviously, the USEPA's acceptance criteria are not based on historical data from PT studies. The USEPA criteria require that this analyte be manufactured at concentrations both above and below 10.0 µg/L and randomly assigned in each study. The failure rate for chloromethane in ERA's WS-87 Study would be 6.67% using ERA's statistically determined regression equation.

The high failure rate causes us to question the appropriateness of the acceptance limit criteria provided in the USEPA Criteria Document. ERA will communicate this seeming disparity in failure rates to NIST/NVLAP. When reviewing each laboratory's performance evaluation for this analyte one must take the above information into consideration.

- Study Discussion: WS-87 Unregulated Volatiles - Fluorotrichloromethane

During the review of the WS-87 Study data ERA discovered a failure rate of 28.6% for fluorotrichloromethane in the Unregulated Volatiles sample. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'.

Per the requirements of ERA's NIST NVLAP PT Provider Accreditation (Lab code 2003860) and the NELAC Standards (July 2003), ERA examined all WS-87 data sets for bimodal distribution and/or situations where results from a given method have disproportionally large failure rates. No method biases or multi-modal distributions were found in the WS-87 fluorotrichloromethane data set. Per the USEPA Criteria Document, the acceptance limits for this compound are ±20% of the assigned value when the concentration is equal to or greater than 10.0 µg/L as was the case with WS-87 and ±40% of the assigned value when the concentration is below 10.0 µg/L. Of the last thirty-five PT studies conducted by ERA, when fluorotrichloromethane has been present at a concentration greater than or equal to 10 µg/L, ERA has witnessed an average failure rate of 23.6%. The average failure rate when fluorotrichloromethane has been present at concentrations less than 10 µg/L is 9.92%. To the best of our knowledge the USEPA has never conducted a study containing fluorotrichloromethane so a failure rate is not available to ERA for comparison. The USEPA criteria require that this analyte be manufactured at concentrations both above and below 10.0 µg/L and randomly assigned in each study. The failure rate for fluorotrichloromethane in ERA's WS-87 Study would be 3.57% using ERA's statistically determined regression equation.

The high failure rate causes us to question the

appropriateness of the acceptance limit criteria provided in the USEPA Criteria Document. ERA will communicate this seeming disparity in failure rates to NIST/NVLAP. When reviewing each laboratory's performance evaluation for this analyte one must take the above information into consideration.

Study Discussion: Hardness - Magnesium

During the review of the magnesium data for the WS-87 Hardness sample ERA observed a failure rate of 30.4%. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'.

Per the requirements of ERA's NIST NVLAP PT Provider Accreditation (Lab code 2003860) and the NELAC Standards (July 2003), ERA examined all WS-87 data sets for bimodal distribution and/or situations where results from a given method have disproportionally large failure rates. No significant method biases or multi-modal distributions were found in the WS-87 magnesium data set. However, we did observe for Standard Method 3500-Mg-E that three data points were reported with all data points failing. Two of the data points were failures high with one failure low. ERA's historical failure rate for magnesium is 14.9%. No concentration bias for high failure rates was determined during the review of the historical failure rate data. The reported study data and 'Not Acceptable' evaluations are both evenly distributed around the mean and assigned value for the WS-87 magnesium study data.

The acceptance criteria posted for proficiency testing providers in the NELAC FOT tables (April 2002) for magnesium is a regression equation that yields limits of 90.7% to 109.7% at 2.79 mg/L, while ERA's historical limits are 87.8% to 114%. The NELAC limits are approximately 3% to 4% tighter than ERA historical limits.

If you have any questions concerning the acceptance criteria or study data please feel free to call Shawn Kassner, Manager Proficiency Testing, or Curtis Wood, Quality Assurance Director, at 1-800.372-0122. If you have any technical questions please feel free to call ERA's Inorganic Department at 1-800-372-0122.

ENVIRONMENTAL
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Study: **WS-87**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Laboratories**

Report Type: **Complete**

Report Method: **Method A**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-87 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Laboratories
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Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/15/03
Study Dates: 10/10/03 - 11/24/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Metals							
1000	Aluminum	µg/L	849	878	789 - 963	Acceptable	EPA 200.7
0140	Antimony †	µg/L		19.2	13.4 - 25.0		
0001	Arsenic †	µg/L		92.3	80.8 - 103		
0002	Barium †	µg/L	697	715	608 - 822	Acceptable	EPA 200.7
0141	Beryllium †	µg/L	3.18	3.51	2.98 - 4.04	Acceptable	EPA 200.7
0226	Boron †	µg/L		807	752 - 889		
0003	Cadmium †	µg/L	24.7	25.5	20.4 - 30.6	Acceptable	EPA 200.7
0004	Chromium †	µg/L	79.1	78.9	67.1 - 90.7	Acceptable	EPA 200.7
0091	Copper †	µg/L	803	804	724 - 884	Acceptable	EPA 200.7
1070	Iron	µg/L	195	194	176 - 214	Acceptable	EPA 200.7
0005	Lead †	µg/L		26.9	18.8 - 35.0		
0236	Manganese †	µg/L	165	163	151 - 172	Acceptable	EPA 200.7
0237	Molybdenum †	µg/L	97.8	100	84.2 - 115	Acceptable	EPA 200.7
0142	Nickel †	µg/L	219	221	188 - 254	Acceptable	EPA 200.7
0007	Selenium †	µg/L		48.7	39.0 - 58.4		
1150	Silver	µg/L	430	400	362 - 439	Acceptable	EPA 200.7
0143	Thallium †	µg/L		5.75	4.03 - 7.48		
1185	Vanadium	µg/L	447	462	430 - 491	Acceptable	EPA 200.7
0239	Zinc †	µg/L	1030	976	897 - 1050	Acceptable	EPA 200.7
Mercury							
0006	Mercury †	µg/L	2.68	2.10	1.47 - 2.73	Acceptable	EPA 200.8
Inorganics							
1575	Chloride	mg/L	63.6	67.0	62.4 - 71.2	Acceptable	EPA 300.0
1610	Conductivity	µmhos/cm	454	452	428 - 476	Acceptable	EPA 120.1
0010	Fluoride †	mg/L	1.82	2.06	1.85 - 2.27	Not Acceptable	EPA 300.0
0009	Nitrate as N †	mg/L	3.99	4.42	3.98 - 4.86	Acceptable	EPA 300.0
1820	Nitrate + Nitrite as N	mg/L	3.99	4.42	3.98 - 4.86	Acceptable	EPA 300.0
1125	Potassium	mg/L	17.1	16.6	15.0 - 18.3	Acceptable	EPA 200.7
0145	Sulfate †	mg/L	11.0	11.4	9.59 - 12.9	Acceptable	EPA 300.0
0024	Total Dissolved Solids at 180°C †	mg/L	335	357	231 - 483	Acceptable	EPA 160.1
TOC							
0263	Total organic carbon †	mg/L	3.77	3.80	3.42 - 4.38	Acceptable	EPA 415.1

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-87 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Laboratories
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/15/03
Study Dates: 10/10/03 - 11/24/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Volatiles							
0039	Benzene †	µg/L	10.5	10.7	8.56 - 12.8	Acceptable	EPA 524.2
0037	Carbon tetrachloride †	µg/L	9.08	7.85	4.71 - 11.0	Acceptable	EPA 524.2
0049	Chlorobenzene †	µg/L	20.4	22.2	17.8 - 26.6	Acceptable	EPA 524.2
0054	1,2-Dichlorobenzene †	µg/L	15.1	14.3	11.4 - 17.2	Acceptable	EPA 524.2
0041	1,4-Dichlorobenzene †	µg/L	7.70	7.15	4.29 - 10.0	Acceptable	EPA 524.2
0035	1,2-Dichloroethane †	µg/L	6.13	7.04	4.22 - 9.86	Acceptable	EPA 524.2
0034	1,1-Dichloroethylene †	µg/L	20.2	17.0	13.6 - 20.4	Acceptable	EPA 524.2
0043	cis-1,2-Dichloroethylene †	µg/L	12.2	11.6	9.28 - 13.9	Acceptable	EPA 524.2
0042	trans-1,2-Dichloroethylene †	µg/L	32.4	30.0	24.0 - 36.0	Acceptable	EPA 524.2
0044	1,2-Dichloropropane †	µg/L	17.4	18.6	14.9 - 22.3	Acceptable	EPA 524.2
0048	Ethylbenzene †	µg/L	18.1	18.3	14.6 - 22.0	Acceptable	EPA 524.2
0055	Methylene chloride †	µg/L	6.22	8.35	5.01 - 11.7	Acceptable	EPA 524.2
0053	Styrene †	µg/L	13.2	14.4	11.5 - 17.3	Acceptable	EPA 524.2
0040	Tetrachloroethylene †	µg/L	17.3	16.5	13.2 - 19.8	Acceptable	EPA 524.2
0047	Toluene †	µg/L	5.13	6.47	3.88 - 9.06	Acceptable	EPA 524.2
0076	1,2,4-Trichlorobenzene †	µg/L	8.81	11.7	9.36 - 14.0	Not Acceptable	EPA 524.2
0036	1,1,1-Trichloroethane †	µg/L	3.56	3.89	2.33 - 5.45	Acceptable	EPA 524.2
0061	1,1,2-Trichloroethane †	µg/L	9.02	10.1	8.08 - 12.1	Acceptable	EPA 524.2
0038	Trichloroethylene †	µg/L	12.7	14.1	11.3 - 16.9	Acceptable	EPA 524.2
0032	Vinyl chloride †	µg/L	17.8	13.7	8.22 - 19.2	Acceptable	EPA 524.2
0090	Xylenes, total †	µg/L	15.5	17.4	13.9 - 20.9	Acceptable	EPA 524.2
Halomethanes							
0019	Bromodichloromethane †	µg/L	21.1	20.0	16.0 - 24.0	Acceptable	EPA 524.2
0018	Bromoform †	µg/L	4.45	4.77	2.86 - 6.68	Acceptable	EPA 524.2
0020	Chlorodibromomethane †	µg/L	29.5	26.8	21.4 - 32.2	Acceptable	EPA 524.2
0017	Chloroform †	µg/L	35.8	30.6	24.5 - 36.7	Acceptable	EPA 524.2
0021	Total Trihalomethanes †	µg/L	90.7	82.2	65.8 - 98.6	Acceptable	EPA 524.2
Unregulated Volatiles							
0067	Bromobenzene †	µg/L	9.76	9.86	5.92 - 13.8	Acceptable	EPA 524.2
0089	Bromochloromethane †	µg/L	13.4	12.4	9.92 - 14.9	Acceptable	EPA 524.2
0069	Bromomethane †	µg/L	3.20	0.00		Not Acceptable	EPA 524.2
0079	n-Butylbenzene †	µg/L	6.32	6.45	3.87 - 9.03	Acceptable	EPA 524.2
0086	sec-Butylbenzene †	µg/L	8.31	9.18	5.51 - 12.9	Acceptable	EPA 524.2
0085	tert-Butylbenzene †	µg/L	19.3	18.1	14.5 - 21.7	Acceptable	EPA 524.2
5000	tert-Butylmethyl ether (MTBE)	µg/L	< 0.5	0.00		Acceptable	EPA 524.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-87 Final Complete Report

Tenkasi S. Viswanathan
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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/15/03
Study Dates: 10/10/03 - 11/24/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Unregulated Volatiles (Cont.)							
0070	Chloroethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0068	Chloromethane †	µg/L	12.4	16.7	13.4 - 20.0	Not Acceptable	EPA 524.2
0071	2-Chlorotoluene †	µg/L	13.4	13.3	10.6 - 16.0	Acceptable	EPA 524.2
0072	4-Chlorotoluene †	µg/L	9.93	9.64	5.78 - 13.5	Acceptable	EPA 524.2
0057	Dibromomethane †	µg/L	8.68	9.70	5.82 - 13.6	Acceptable	EPA 524.2
0066	1,3-Dichlorobenzene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0088	Dichlorodifluoromethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0056	1,1-Dichloroethane †	µg/L	11.9	11.1	8.88 - 13.3	Acceptable	EPA 524.2
0059	1,3-Dichloropropane †	µg/L	11.4	10.6	8.48 - 12.7	Acceptable	EPA 524.2
0060	2,2-Dichloropropane †	µg/L	9.15	8.69	5.21 - 12.2	Acceptable	EPA 524.2
0058	1,1-Dichloropropene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0152	cis-1,3-Dichloropropene †	µg/L	10.9	12.9	10.3 - 15.5	Acceptable	EPA 524.2
0153	trans-1,3-Dichloropropene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0087	Fluorotrichloromethane †	µg/L	19.0	16.9	13.5 - 20.3	Acceptable	EPA 524.2
0081	Hexachlorobutadiene †	µg/L	12.7	14.0	11.2 - 16.8	Acceptable	EPA 524.2
0084	Isopropylbenzene †	µg/L	10.8	11.2	8.96 - 13.4	Acceptable	EPA 524.2
0083	4-Isopropyltoluene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0078	n-Propylbenzene †	µg/L	16.8	16.0	12.8 - 19.2	Acceptable	EPA 524.2
0063	1,1,1,2-Tetrachloroethane †	µg/L	7.77	7.90	4.74 - 11.1	Acceptable	EPA 524.2
0065	1,1,2,2-Tetrachloroethane †	µg/L	12.6	12.7	10.2 - 15.2	Acceptable	EPA 524.2
0077	1,2,3-Trichlorobenzene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0064	1,2,3-Trichloropropane †	µg/L	9.44	11.7	9.36 - 14.0	Acceptable	EPA 524.2
0075	1,2,4-Trimethylbenzene †	µg/L	3.47	3.47	2.08 - 4.86	Acceptable	EPA 524.2
0082	1,3,5-Trimethylbenzene †	µg/L	5.44	5.54	3.32 - 7.76	Acceptable	EPA 524.2
Regulated Pesticides							
0093	Alachlor †	µg/L	17.9	16.6	9.13 - 24.1	Acceptable	EPA 525.2
0256	Aldrin †	µg/L	1.50	1.68	0.713 - 2.11	Acceptable	EPA 508.1
0094	Atrazine †	µg/L	5.1	5.95	3.27 - 8.63	Acceptable	EPA 525.2
7160	Butachlor	µg/L		16.6	9.60 - 20.6		
7410	Diazinon	µg/L		0.00			
0258	Dieldrin †	µg/L	1.30	1.54	1.08 - 1.93	Acceptable	EPA 508.1
0011	Endrin †	µg/L	0.838	1.02	0.714 - 1.33	Acceptable	EPA 508.1
0095	Heptachlor †	µg/L	4.16	4.65	2.56 - 6.74	Acceptable	EPA 508.1
0096	Heptachlor epoxide (beta) †	µg/L	1.92	2.29	1.26 - 3.32	Acceptable	EPA 508.1
0172	Hexachlorobenzene †	µg/L	2.60	2.95	1.36 - 3.74	Acceptable	EPA 525.2
0112	Hexachlorocyclopentadiene †	µg/L	2.61	3.17	0.121 - 4.05	Acceptable	EPA 525.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

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WS-87 Final Complete Report

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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/15/03
Study Dates: 10/10/03 - 11/24/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Pesticides (Cont.)							
0012	gamma-BHC (Lindane) †	µg/L	1.28	1.55	0.853 - 2.25	Acceptable	EPA 508.1
0013	Methoxychlor †	µg/L	13.3	14.9	8.20 - 21.6	Acceptable	EPA 508.1
7835	Metolachlor	µg/L		0.00			
7845	Metribuzin	µg/L		14.9	10.1 - 17.9		
7875	Molinate (ordram)	µg/L		10.7	5.06 - 15.3		
8035	Prometon	µg/L		0.00			
0259	Propachlor †	µg/L		2.40	1.43 - 3.38		
0113	Simazine †	µg/L	11.9	6.61	1.63 - 10.6	Not Acceptable	EPA 525.2
8220	Thiobencarb	µg/L		0.00			
0244	Trifluralin †	µg/L		2.42	1.02 - 3.11		
Toxaphene							
0014	Toxaphene †	µg/L	8.88	9.25	5.09 - 13.4	Acceptable	EPA 508.1
Chlordane							
0097	Chlordane, technical †	µg/L	15.7	18.2	10.0 - 26.4	Acceptable	EPA 508.1
EDB/DBCP							
0045	Dibromochloropropane (DBCP) †	µg/L	0.187	0.187	0.112 - 0.262	Acceptable	EPA 504.1
0046	Ethylene Dibromide (EDB) †	µg/L	1.65	1.62	0.972 - 2.27	Acceptable	EPA 504.1
5180	1,2,3-Trichloropropane	µg/L	1.64	1.76	0.887 - 2.45	Acceptable	EPA 504.1
Regulated Semivolatiles #1							
5500	Acenaphthene	µg/L	4.12	5.35	2.36 - 7.12	Acceptable	EPA 525.2
5505	Acenaphthylene	µg/L	< 0.20	0.00		Acceptable	EPA 525.2
5555	Anthracene	µg/L	0.79	0.936	0.329 - 1.28	Acceptable	EPA 525.2
5575	Benzo(a)anthracene	µg/L	< 0.20	0.00		Acceptable	EPA 525.2
5585	Benzo(b)fluoranthene	µg/L	< 0.20	0.00		Acceptable	EPA 525.2
5600	Benzo(k)fluoranthene	µg/L	< 0.20	0.00		Acceptable	EPA 525.2
5590	Benzo(g,h,i)perylene	µg/L	< 0.20	0.00		Acceptable	EPA 525.2
0122	Benzo(a)pyrene †	µg/L	0.57	0.632	0.168 - 0.844	Acceptable	EPA 525.2
5670	Butylbenzylphthalate	µg/L	< 5.00	0.00		Acceptable	EPA 525.2
5855	Chrysene	µg/L	< 0.20	0.00		Acceptable	EPA 525.2
5895	Dibenz(a,h)anthracene	µg/L	< 0.20	0.00		Acceptable	EPA 525.2
5925	Di-n-butylphthalate	µg/L	< 5.00	0.00		Acceptable	EPA 525.2
6070	Diethylphthalate	µg/L	23.1	25.6	7.96 - 37.9	Acceptable	EPA 525.2
6135	Dimethylphthalate	µg/L	34.7	40.2	15.8 - 56.3	Acceptable	EPA 525.2
6200	Di-n-octylphthalate	µg/L	< 5.00	0.00		Acceptable	EPA 525.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-87 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Laboratories
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/15/03
Study Dates: 10/10/03 - 11/24/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Semivolatiles #1 (Cont.)							
0134	bis(2-Ethylhexyl)adipate †	µg/L	38.6	44.7	18.8 - 64.3	Acceptable	EPA 525.2
0136	bis(2-Ethylhexyl)phthalate †	µg/L	19	26.5	11.1 - 41.2	Acceptable	EPA 525.2
6265	Fluoranthene	µg/L	1.42	1.79	0.882 - 2.38	Acceptable	EPA 525.2
6270	Fluorene	µg/L	< 0.20	0.00		Acceptable	EPA 525.2
6315	Indeno(1,2,3-cd)pyrene	µg/L	< 0.20	0.00		Acceptable	EPA 525.2
5005	Naphthalene	µg/L	< 0.20	0.00		Acceptable	EPA 525.2
6615	Phenanthrene	µg/L	< 0.20	0.00		Acceptable	EPA 525.2
6665	Pyrene	µg/L	< 0.20	0.00		Acceptable	EPA 525.2
Regulated Herbicides							
0262	Acifluorfen †	µg/L		26.0	7.60 - 38.7		
8530	Bentazon	µg/L		0.00			
8540	Chloramben	µg/L		0.00			
0015	2,4-D †	µg/L	29.7	135	67.5 - 203	Not Acceptable	EPA 515.4
8560	2,4-DB	µg/L	54.1	53.9	20.6 - 77.4	Acceptable	EPA 515.4
8550	Dacthal diacid (DCPA)	µg/L		0.00			
0115	Dalapon †	µg/L	1.44	139	0.00 - 188	Acceptable	EPA 515.4
0247	Dicamba †	µg/L	6.36	28.4	7.90 - 40.0	Not Acceptable	EPA 515.4
8600	3,5-Dichlorobenzoic acid	µg/L		0.00			
8605	Dichlorprop	µg/L	< 1.00	0.00		Acceptable	EPA 515.4
0116	Dinoseb †	µg/L	< 1.00	25.9	0.577 - 38.8	Not Acceptable	EPA 515.4
6500	4-Nitrophenol	µg/L		95.9	0.00 - 195		
0102	Pentachlorophenol †	µg/L	55.0	53.4	26.7 - 80.1	Acceptable	EPA 525.2
0117	Picloram †	µg/L		42.6	0.0514 - 57.6		
8655	2,4,5-T	µg/L	< 1.00	0.00		Acceptable	EPA 515.4
0016	2,4,5-TP (Silvex) †	µg/L	8.10	15.4	7.70 - 23.1	Acceptable	EPA 515.4

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL
RESOURCE ASSOCIATES®

Study: **WS-87**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Laboratories**

Report Type: **Complete**

Report Method: **Method B**

ACCREDITED
Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP
Lab Code: 200386-0

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-87 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Laboratories
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/15/03
Study Dates: 10/10/03 - 11/24/03

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Metals							
1000	Aluminum	µg/L	836	878	789 - 963	Acceptable	EPA 200.8
0140	Antimony †	µg/L	17.8	19.2	13.4 - 25.0	Acceptable	EPA 200.8
0001	Arsenic †	µg/L	90.4	92.3	80.8 - 103	Acceptable	EPA 200.8
0002	Barium †	µg/L	724	715	608 - 822	Acceptable	EPA 200.8
0141	Beryllium †	µg/L	3.51	3.51	2.98 - 4.04	Acceptable	EPA 200.8
0226	Boron †	µg/L	740	807	752 - 889	Not Acceptable	EPA 200.8
0003	Cadmium †	µg/L	23.7	25.5	20.4 - 30.6	Acceptable	EPA 200.8
0004	Chromium †	µg/L	76.8	78.9	67.1 - 90.7	Acceptable	EPA 200.8
0091	Copper †	µg/L		804	724 - 884		
1070	Iron	µg/L		194	176 - 214		
0005	Lead †	µg/L	26.6	26.9	18.8 - 35.0	Acceptable	EPA 200.8
0236	Manganese †	µg/L	159	163	151 - 172	Acceptable	EPA 200.8
0237	Molybdenum †	µg/L	95.0	100	84.2 - 115	Acceptable	EPA 200.8
0142	Nickel †	µg/L	194	221	188 - 254	Acceptable	EPA 200.8
0007	Selenium †	µg/L	48.8	48.7	39.0 - 58.4	Acceptable	EPA 200.8
1150	Silver	µg/L		400	362 - 439		
0143	Thallium †	µg/L	5.63	5.75	4.03 - 7.48	Acceptable	EPA 200.8
1185	Vanadium	µg/L		462	430 - 491		
0239	Zinc †	µg/L	972	976	897 - 1050	Acceptable	EPA 200.8
Mercury							
0006	Mercury †	µg/L	2.79	2.10	1.47 - 2.73	Not Acceptable	EPA 245.1
Inorganics							
1575	Chloride	mg/L	70.9	67.0	62.4 - 71.2	Acceptable	EPA 325.3
1610	Conductivity	µmhos/cm		452	428 - 476		
0010	Fluoride †	mg/L		2.06	1.85 - 2.27		
0009	Nitrate as N †	mg/L	3.84	4.42	3.98 - 4.86	Not Acceptable	EPA 353.2
1820	Nitrate + Nitrite as N	mg/L	3.84	4.42	3.98 - 4.86	Not Acceptable	EPA 353.2
1125	Potassium	mg/L		16.6	15.0 - 18.3		
0145	Sulfate †	mg/L	11.3	11.4	9.59 - 12.9	Acceptable	EPA 375.4
0024	Total Dissolved Solids at 180°C †	mg/L		357	231 - 483		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-89 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 **EPA ID: KS00902**

Report Issued: 02/16/04
Study Dates: 12/15/03 - 01/29/04

WS Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Values are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, and the National Environmental Laboratory Accreditation Conference (NELAC) program criteria as applicable. A parameter not added to the standard is given an Assigned Value of "0" per the guidelines contained in the USEPA's Criteria Document and NELAC standards.

The Acceptance Limits are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, the NELAC PT program criteria or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside of the Acceptance Limits.
No Evaluation	=	Reported Value cannot be evaluated.

The Method Description is the method the laboratory reported to ERA.

WS Study Discussion:

ERA's WatR™ Supply Proficiency Testing Study, WS-89, has been reviewed by ERA Senior Management and certified compliant with the requirements of the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document (December 1998), the NELAC Standards Chapter 2 and Associated Appendices, applicable state PT programs, and those contained in the National Institute of Standards and Technology NVLAP Handbooks 150 and 150-19. ERA is a NIST NVLAP accredited PT Provider (Lab Code 200386-0).

This report contains data that are not covered by the NVLAP accreditation.

ERA's WatR™ Supply Study, WS-89, standards were examined for any anomalies. A full review of all accuracy verification, homogeneity and stability (VHS) data was completed. All analytical verification data for all analytes in the WS-89 standards met the acceptance criteria contained in the USEPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, and the National Voluntary Laboratory Accreditation Program Handbook 150-19 for Chemical Calibration for Providers of Proficiency Testing.

The data submitted by participating laboratories was also examined for study anomalies. There were no anomalies found during the review of the study data.

WatR™ Supply Study, WS-89, reports shall not be reproduced except in their entirety and not without the permission of the participating laboratories. The report must not be used by the participating laboratories to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding ERA's WatR™ Supply Proficiency Testing Study, WS-89, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

Study: **WS-89**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Laboratories, Inc.**

Report Type: **Complete**

Report Method: **Method A**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-89 Final Complete Report

Tenkasi S. Viswanathan
Analytical Management Laboratories, Inc.
15130 S Keeler
Suite B
Olathe, KS 66062
913-829-0101

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 02/16/04
Study Dates: 12/15/03 - 01/29/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Mercury							
0006	Mercury †	µg/L	7.83	7.98	5.59 - 10.4	Acceptable	EPA 200.8
Inorganics							
1575	Chloride	mg/L		68.9	64.3 - 73.2		
1610	Conductivity	µmhos/cm	488	491	465 - 517	Acceptable	EPA 120.1
0010	Fluoride †	mg/L	1.79	1.89	1.70 - 2.08	Acceptable	EPA 300.0
0009	Nitrate as N †	mg/L	5.25	5.58	5.02 - 6.14	Acceptable	EPA 300.0
1820	Nitrate + Nitrite as N	mg/L	5.25	5.58	5.02 - 6.14	Acceptable	EPA 300.0
1125	Potassium	mg/L		19.5	17.7 - 21.4		
0145	Sulfate †	mg/L		28.0	24.7 - 31.0		
0024	Total Dissolved Solids at 180°C †	mg/L		359	232 - 486		
TOC							
0263	Total organic carbon †	mg/L	2.65	2.44	2.13 - 2.91	Acceptable	EPA 415.1

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-89 Final Complete Report

Tenkasi S. Viswanathan
Analytical Management Laboratories, Inc.
15130 S Keeler
Suite B
Olathe, KS 66062
913-829-0101

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 02/16/04
Study Dates: 12/15/03 - 01/29/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Volatiles							
0039	Benzene †	µg/L		14.9	11.9 - 17.9		
0037	Carbon tetrachloride †	µg/L		11.8	9.44 - 14.2		
0049	Chlorobenzene †	µg/L		29.1	23.3 - 34.9		
0054	1,2-Dichlorobenzene †	µg/L		9.40	5.64 - 13.2		
0041	1,4-Dichlorobenzene †	µg/L		7.71	4.63 - 10.8		
0035	1,2-Dichloroethane †	µg/L		15.8	12.6 - 19.0		
0034	1,1-Dichloroethylene †	µg/L		2.28	1.37 - 3.19		
0043	cis-1,2-Dichloroethylene †	µg/L		16.7	13.4 - 20.0		
0042	trans-1,2-Dichloroethylene †	µg/L		30.6	24.5 - 36.7		
0044	1,2-Dichloropropane †	µg/L		15.0	12.0 - 18.0		
0048	Ethylbenzene †	µg/L		2.80	1.68 - 3.92		
0055	Methylene chloride †	µg/L		11.3	9.04 - 13.6		
0053	Styrene †	µg/L		17.3	13.8 - 20.8		
0040	Tetrachloroethylene †	µg/L		16.2	13.0 - 19.4		
0047	Toluene †	µg/L		8.77	5.26 - 12.3		
0076	1,2,4-Trichlorobenzene †	µg/L	19.6	18.4	14.7 - 22.1	Acceptable	EPA 524.2
0036	1,1,1-Trichloroethane †	µg/L		17.5	14.0 - 21.0		
0061	1,1,2-Trichloroethane †	µg/L		3.04	1.82 - 4.26		
0038	Trichloroethylene †	µg/L		18.9	15.1 - 22.7		
0032	Vinyl chloride †	µg/L		18.8	11.3 - 26.3		
0090	Xylenes, total †	µg/L		25.7	20.6 - 30.8		
Unregulated Volatiles							
0067	Bromobenzene †	µg/L		14.6	11.7 - 17.5		
0089	Bromochloromethane †	µg/L		6.74	4.04 - 9.44		
0069	Bromomethane †	µg/L	10.6	8.40	5.04 - 11.8	Acceptable	EPA 524.2
0079	n-Butylbenzene †	µg/L		0.00			
0086	sec-Butylbenzene †	µg/L		16.8	13.4 - 20.2		
0085	tert-Butylbenzene †	µg/L		0.00			
5000	tert-Butylmethyl ether (MTBE)	µg/L		0.00			
0070	Chloroethane †	µg/L		0.00			
0068	Chloromethane †	µg/L		19.6	15.7 - 23.5		
0071	2-Chlorotoluene †	µg/L		0.00			
0072	4-Chlorotoluene †	µg/L		14.5	11.6 - 17.4		
0057	Dibromomethane †	µg/L		19.6	15.7 - 23.5		
0066	1,3-Dichlorobenzene †	µg/L		19.3	15.4 - 23.2		
0088	Dichlorodifluoromethane †	µg/L		0.00			

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-89 Final Complete Report

Tenkasi S. Viswanathan
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15130 S Keeler
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913-829-0101

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 02/16/04
Study Dates: 12/15/03 - 01/29/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Unregulated Volatiles (Cont.)							
0056	1,1-Dichloroethane †	µg/L		3.95	2.37 - 5.53		
0059	1,3-Dichloropropane †	µg/L		19.6	15.7 - 23.5		
0060	2,2-Dichloropropane †	µg/L		14.9	11.9 - 17.9		
0058	1,1-Dichloropropene †	µg/L		2.29	1.37 - 3.21		
0152	cis-1,3-Dichloropropene †	µg/L		2.80	1.68 - 3.92		
0153	trans-1,3-Dichloropropene †	µg/L		2.92	1.75 - 4.09		
0087	Fluorotrichloromethane †	µg/L		0.00			
0081	Hexachlorobutadiene †	µg/L		0.00			
0084	Isopropylbenzene †	µg/L		16.5	13.2 - 19.8		
0083	4-Isopropyltoluene †	µg/L		0.00			
0078	n-Propylbenzene †	µg/L		2.80	1.68 - 3.92		
0063	1,1,1,2-Tetrachloroethane †	µg/L		5.88	3.53 - 8.23		
0065	1,1,2,2-Tetrachloroethane †	µg/L		14.7	11.8 - 17.6		
0077	1,2,3-Trichlorobenzene †	µg/L		7.45	4.47 - 10.4		
0064	1,2,3-Trichloropropane †	µg/L		19.6	15.7 - 23.5		
0075	1,2,4-Trimethylbenzene †	µg/L		0.00			
0082	1,3,5-Trimethylbenzene †	µg/L		6.98	4.19 - 9.77		
Regulated Herbicides							
0262	Acifluorfen †	µg/L	20.1	23.9	6.11 - 36.5	Acceptable	EPA 515.4
8530	Bentazon	µg/L	< 1.00	0.00		Acceptable	EPA 515.4
8540	Chloramben	µg/L		42.9	0.0806 - 53.6		
0015	2,4-D †	µg/L	64.6	122	61.0 - 183	Acceptable	EPA 515.4
8560	2,4-DB	µg/L	< 1.00	0.00		Acceptable	EPA 515.4
8550	Dacthal diacid (DCPA)	µg/L	< 1.00	0.00		Acceptable	EPA 515.4
0115	Dalapon †	µg/L		124	0.00 - 168		
0247	Dicamba †	µg/L	68.1	60.4	16.0 - 83.8	Acceptable	EPA 515.4
8600	3,5-Dichlorobenzoic acid	µg/L	< 1.00	0.00		Acceptable	EPA 515.4
8605	Dichlorprop	µg/L		34.6	9.86 - 52.9		
0116	Dinoseb †	µg/L	18.2	36.9	1.33 - 55.5	Acceptable	EPA 515.4
6500	4-Nitrophenol	µg/L	< 1.00	0.00		Acceptable	EPA 515.4
0102	Pentachlorophenol †	µg/L	70	95.6	47.8 - 143	Acceptable	EPA 515.4
0117	Picloram †	µg/L	5.8	27.0	0.357 - 37.9	Acceptable	EPA 515.4
8655	2,4,5-T	µg/L		80.5	17.8 - 126		
0016	2,4,5-TP (Silvex) †	µg/L		20.5	10.3 - 30.8		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

Study: **WS-89**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Laboratories, Inc.**

Report Type: **Complete**

Report Method: **Method B**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-89 Final Complete Report

Tenkasi S. Viswanathan
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913-829-0101

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 02/16/04
Study Dates: 12/15/03 - 01/29/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Mercury							
0006	Mercury †	µg/L	8.70	7.98	5.59 - 10.4	Acceptable	EPA 245.1
Inorganics							
1575	Chloride	mg/L		68.9	64.3 - 73.2		
1610	Conductivity	µmhos/cm		491	465 - 517		
0010	Fluoride †	mg/L		1.89	1.70 - 2.08		
0009	Nitrate as N †	mg/L	5.19	5.58	5.02 - 6.14	Acceptable	EPA 353.2
1820	Nitrate + Nitrite as N	mg/L	5.20	5.58	5.02 - 6.14	Acceptable	EPA 353.2
1125	Potassium	mg/L		19.5	17.7 - 21.4		
0145	Sulfate †	mg/L		28.0	24.7 - 31.0		
0024	Total Dissolved Solids at 180°C †	mg/L		359	232 - 486		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-90 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 **EPA ID: KS00902**

Report Issued: 03/18/04
Study Dates: 01/12/04 - 02/26/04

WS Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Values are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, and the National Environmental Laboratory Accreditation Conference (NELAC) program criteria as applicable. A parameter not added to the standard is given an Assigned Value of "0" per the guidelines contained in the USEPA's Criteria Document and NELAC standards.

The Acceptance Limits are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, the NELAC PT program criteria or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside of the Acceptance Limits.
No Evaluation	=	Reported Value cannot be evaluated.

The Method Description is the method the laboratory reported to ERA.

WS Study Discussion:

ERA's WatR™ Supply Proficiency Testing Study, WS-90, has been reviewed by ERA Senior Management and certified compliant with the requirements of the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document (December 1998), the NELAC Standards Chapter 2 and Associated Appendices, applicable state PT programs, and those contained in the National Institute of Standards and Technology NVLAP Handbooks 150 and 150-19. ERA is a NIST NVLAP accredited PT Provider (Lab Code 200386-0).

This report contains data that are not covered by the NVLAP accreditation.

ERA's WatR™ Supply Study, WS-90, standards were examined for any anomalies. A full review of all accuracy verification, homogeneity and stability (VHS) data was completed. All analytical verification data for all analytes in the WS-90 standards met the acceptance criteria contained in the USEPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, and the National Voluntary Laboratory Accreditation Program Handbook 150-19 for Chemical Calibration for Providers of Proficiency Testing.

The data submitted by participating laboratories was also examined for study anomalies. There were no anomalies found during the review of the study data.

WatR™ Supply Study, WS-90, reports shall not be reproduced except in their entirety and not without the permission of the participating laboratories. The report must not be used by the participating laboratories to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding ERA's WatR™ Supply Proficiency Testing Study, WS-90, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

Study: **WS-90**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method A**

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-90 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 03/18/04
Study Dates: 01/12/04 - 02/26/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Mercury							
0006	Mercury †	µg/L	2.22	2.39	1.67 - 3.11	Acceptable	EPA 200.8
Inorganics							
1575	Chloride	mg/L		130	122 - 138		
1610	Conductivity	µmhos/cm	617	615	582 - 648	Acceptable	EPA 120.1
0010	Fluoride †	mg/L	4.90	5.02	4.52 - 5.52	Acceptable	EPA 300.0
0009	Nitrate as N †	mg/L	6.97	7.18	6.46 - 7.90	Acceptable	EPA 300.0
1820	Nitrate + Nitrite as N	mg/L	6.97	7.18	6.46 - 7.90	Acceptable	EPA 300.0
1125	Potassium	mg/L		30.4	27.7 - 33.3		
0145	Sulfate †	mg/L	14.8	15.4	13.2 - 17.3	Acceptable	EPA 300.0
0024	Total Dissolved Solids at 180°C †	mg/L	422	358	231 - 485	Acceptable	EPA 160.1
Total Organic Carbon							
0263	Total organic carbon †	mg/L	4.71	4.39	3.98 - 5.02	Acceptable	EPA 415.1

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-90 Final Complete Report

Tenkasi S. Viswanathan
QA Director
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15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 03/18/04
Study Dates: 01/12/04 - 02/26/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Volatiles							
0039	Benzene †	µg/L		10.7	8.56 - 12.8		
0037	Carbon tetrachloride †	µg/L		7.61	4.57 - 10.7		
0049	Chlorobenzene †	µg/L		22.0	17.6 - 26.4		
0054	1,2-Dichlorobenzene †	µg/L		15.0	12.0 - 18.0		
0041	1,4-Dichlorobenzene †	µg/L		4.06	2.44 - 5.68		
0035	1,2-Dichloroethane †	µg/L		7.01	4.21 - 9.81		
0034	1,1-Dichloroethylene †	µg/L		16.9	13.5 - 20.3		
0043	cis-1,2-Dichloroethylene †	µg/L		31.5	25.2 - 37.8		
0042	trans-1,2-Dichloroethylene †	µg/L		20.3	16.2 - 24.4		
0044	1,2-Dichloropropane †	µg/L		9.62	5.77 - 13.5		
0048	Ethylbenzene †	µg/L		18.3	14.6 - 22.0		
0055	Methylene chloride †	µg/L		8.32	4.99 - 11.6		
0053	Styrene †	µg/L		14.2	11.4 - 17.0		
0040	Tetrachloroethylene †	µg/L		11.7	9.36 - 14.0		
0047	Toluene †	µg/L		6.40	3.84 - 8.96		
0076	1,2,4-Trichlorobenzene †	µg/L	10.7	11.5	9.20 - 13.8	Acceptable	EPA 524.2
0036	1,1,1-Trichloroethane †	µg/L		3.89	2.33 - 5.45		
0061	1,1,2-Trichloroethane †	µg/L		9.87	5.92 - 13.8		
0038	Trichloroethylene †	µg/L		14.2	11.4 - 17.0		
0032	Vinyl chloride †	µg/L		2.75	1.65 - 3.85		
0090	Xylenes, total †	µg/L		17.4	13.9 - 20.9		
Unregulated Volatiles							
0067	Bromobenzene †	µg/L		7.39	4.43 - 10.3		
0089	Bromochloromethane †	µg/L		4.78	2.87 - 6.69		
0069	Bromomethane †	µg/L		0.00			
0079	n-Butylbenzene †	µg/L		9.40	5.64 - 13.2		
0086	sec-Butylbenzene †	µg/L	9.32	9.72	5.83 - 13.6	Acceptable	EPA 524.2
0085	tert-Butylbenzene †	µg/L		0.00			
5000	tert-Butylmethyl ether (MTBE)	µg/L		10.6	7.74 - 13.8		
0070	Chloroethane †	µg/L		12.5	10.0 - 15.0		
0068	Chloromethane †	µg/L	8.13	7.50	4.50 - 10.5	Acceptable	EPA 524.2
0071	2-Chlorotoluene †	µg/L		0.00			
0072	4-Chlorotoluene †	µg/L		12.4	9.92 - 14.9		
0057	Dibromomethane †	µg/L		6.38	3.83 - 8.93		
0066	1,3-Dichlorobenzene †	µg/L		16.5	13.2 - 19.8		
0088	Dichlorodifluoromethane †	µg/L		0.00			

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-90 Final Complete Report

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Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 03/18/04
Study Dates: 01/12/04 - 02/26/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Unregulated Volatiles (Cont.)							
0056	1,1-Dichloroethane †	µg/L		6.75	4.05 - 9.45		
0059	1,3-Dichloropropane †	µg/L		15.7	12.6 - 18.8		
0060	2,2-Dichloropropane †	µg/L		10.6	8.48 - 12.7		
0058	1,1-Dichloropropene †	µg/L		7.24	4.34 - 10.1		
0152	cis-1,3-Dichloropropene †	µg/L		7.15	4.29 - 10.0		
0153	trans-1,3-Dichloropropene †	µg/L		13.1	10.5 - 15.7		
0087	Fluorotrichloromethane †	µg/L		2.60	1.56 - 3.64		
0081	Hexachlorobutadiene †	µg/L		0.00			
0084	Isopropylbenzene †	µg/L		9.68	5.81 - 13.6		
0083	4-Isopropyltoluene †	µg/L		0.00			
0078	n-Propylbenzene †	µg/L		12.3	9.84 - 14.8		
0063	1,1,1,2-Tetrachloroethane †	µg/L		0.00			
0065	1,1,2,2-Tetrachloroethane †	µg/L		17.8	14.2 - 21.4		
0077	1,2,3-Trichlorobenzene †	µg/L	15.1	16.7	13.4 - 20.0	Acceptable	EPA 524.2
0064	1,2,3-Trichloropropane †	µg/L		11.8	9.44 - 14.2		
0075	1,2,4-Trimethylbenzene †	µg/L		17.3	13.8 - 20.8		
0082	1,3,5-Trimethylbenzene †	µg/L		9.77	5.86 - 13.7		
Regulated Herbicides							
0262	Acifluorfen †	µg/L	15.4	43.2	19.8 - 57.0	Not Acceptable	EPA 515.4
8530	Bentazon	µg/L	< 1	0.00		Acceptable	EPA 515.4
8540	Chloramben	µg/L		0.00			
0015	2,4-D †	µg/L	31.0	47.5	23.8 - 71.3	Acceptable	EPA 515.4
8560	2,4-DB	µg/L	< 1	0.00		Acceptable	EPA 515.4
8550	Dacthal diacid (DCPA)	µg/L	< 1	0.00		Acceptable	EPA 515.4
0115	Dalapon †	µg/L	0.26	137	0.00 - 185	Acceptable	EPA 515.4
0247	Dicamba †	µg/L	35.8	87.1	22.8 - 120	Acceptable	EPA 515.4
8600	3,5-Dichlorobenzoic acid	µg/L	< 1	0.00		Acceptable	EPA 515.4
8605	Dichlorprop	µg/L	< 1	0.00		Acceptable	EPA 515.4
0116	Dinoseb †	µg/L	7.05	8.47	0.00 - 12.5	Acceptable	EPA 515.4
6500	4-Nitrophenol	µg/L	40.7	136	0.00 - 234	Acceptable	EPA 515.4
0102	Pentachlorophenol †	µg/L	29.6	38.0	19.0 - 57.0	Acceptable	EPA 515.4
0117	Picloram †	µg/L	3.0	45.7	0.00 - 61.6	Acceptable	EPA 515.4
8655	2,4,5-T	µg/L		113	28.3 - 168		
0016	2,4,5-TP (Silvex) †	µg/L	11.7	13.2	6.60 - 19.8	Acceptable	EPA 515.4

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-90 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 03/18/04
Study Dates: 01/12/04 - 02/26/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Perchlorate							
1895	Perchlorate	µg/L	10.6	5.48	4.41 - 6.08	Not Acceptable	EPA 314

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL
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Study: **WS-90**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method B**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-90 Final Complete Report

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QA Director
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Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 03/18/04
Study Dates: 01/12/04 - 02/26/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Mercury							
0006	Mercury †	µg/L	2.58	2.39	1.67 - 3.11	Acceptable	EPA 245.1
Inorganics							
1575	Chloride	mg/L		130	122 - 138		
1610	Conductivity	µmhos/cm		615	582 - 648		
0010	Fluoride †	mg/L		5.02	4.52 - 5.52		
0009	Nitrate as N †	mg/L	7.13	7.18	6.46 - 7.90	Acceptable	EPA 353.2
1820	Nitrate + Nitrite as N	mg/L	7.12	7.18	6.46 - 7.90	Acceptable	EPA 353.2
1125	Potassium	mg/L		30.4	27.7 - 33.3		
0145	Sulfate †	mg/L	15.4	15.4	13.2 - 17.3	Acceptable	EPA 375.4
0024	Total Dissolved Solids at 180°C †	mg/L		358	231 - 485		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL
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The Industry Standard™

June 17, 2004

Tenkasi Viswanathan
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

Enclosed is your final report for ERA's WatR™ Supply Proficiency Testing (PT) Study, WS-93. Your final report includes an evaluation of all results submitted by your laboratory to ERA. Attached is a table listing which regulatory agencies have been sent a copy of your final results and the report type received by those agencies.

Data Evaluation Protocols

ERA's WS-93 PT standards contain every analyte included in every state and national PT program. All analytes have been evaluated using the following tiered approach. If an analyte is listed in the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, the evaluation was completed by comparing the reported result to the acceptance limits generated using the EPA's National Standards. If the analyte is not included in the EPA's National Standards, but is included in the National Environmental Laboratory Accreditation Conference (NELAC) PT Field of Testing list (June 2000), the evaluation has been completed by comparing the reported result to the acceptance limits generated using the evaluation criteria contained in the NELAC standards. If the analyte is not included in either national PT program, the reported result has been evaluated using the procedures outlined in ERA's Standard Operating Procedure for the Generation of Performance Acceptance Limits (SOP 0260).

Corrective Action Help

As part of your accreditation(s), you may be required to identify the root cause of any "Not Acceptable" results, implement the necessary corrective actions, and then satisfy your PT requirements by participating in a Supplemental (QuiK™ Response) or future ERA PT study. If you need help, ERA's technical staff is available to help your laboratory resolve any technical issues that may be impairing your PT performance and possibly affecting your routine data quality. Our laboratory and technical staff have well over three hundred years of collective experience in performing the full range of environmental analyses. As part of our technical support, ERA offers QC samples that can be helpful in helping you work through your technical issues.

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Thank you for your participation in ERA's WatR™ Supply Proficiency Testing Study, WS-93. If you have any questions, please contact myself, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

Sincerely,

Shawn Kassner
Proficiency Testing Manager

attachments
smk

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

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Regulatory Agencies	Agency Requested Report Type	Agency Lab ID	Contact
EPA Region 4	Complete Report		Marilyn Thornton
Florida	Complete Report	E87892	Steve Arms
Kansas	Complete Report	E-10254	Maren Farr
North Carolina (WS)	Complete Report	627	Mike King
Oklahoma	Complete Report		David Caldwell
South Carolina	Complete Report	76003	Connie Turner

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The Industry Standard™

June 17, 2004

Tenkasi Viswanathan
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

In my role as ERA's Quality Assurance Director, I have independently reviewed all aspects of ERA's WatR™ Supply Proficiency Testing Study, WS-93, for compliance with all USEPA, NELAC, NIST NVLAP, and all state technical and program requirements in effect during this study, as well as those of our ISO 9001 Registered Quality System.

All aspects of ERA's WS-93 Study, from standard manufacture to final report generation, were completed by ERA in accordance with the "National Standards for Water Proficiency Testing Studies Criteria Document", USEPA December 30, 1998. ERA has reviewed all of the data that is contained in this report and has made every possible effort to make it complete, accurate and compliant. However, if you find anything in your report that you feel is incomplete, inaccurate or have any quality-related issues, please call me directly at 1-800-372-0122. As required by ERA Standard Operating Procedure for Handling Product and Service Problems (SOP 0150, Rev.7.0), we will initiate an internal investigation and take corrective action as appropriate.

On behalf of ERA, thank you again for your participation in WS-93.

Sincerely,

Curtis J. Wood
Quality Assurance Director

WS-93 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 EPA ID: KS00902

Report Issued: 06/17/04
Study Dates: 04/12/04 - 05/27/04

WS Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Values are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, and the National Environmental Laboratory Accreditation Conference (NELAC) program criteria as applicable. A parameter not added to the standard is given an Assigned Value of "0" per the guidelines contained in the USEPA's Criteria Document and NELAC standards.

The Acceptance Limits are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, the NELAC PT program criteria or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside of the Acceptance Limits.
No Evaluation	=	Reported Value cannot be evaluated.

The Method Description is the method the laboratory reported to ERA.

WS Study Discussion:

ERA's WatR™ Supply Proficiency Testing Study, WS-93, has been reviewed by ERA Senior Management and certified compliant with the requirements of the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document (December 1998), the NELAC Standards Chapter 2 and Associated Appendices, applicable state PT programs, and those contained in the National Institute of Standards and Technology NVLAP Handbooks 150 and 150-19. ERA is a NIST NVLAP accredited PT Provider (Lab Code 200386-0).

This report contains data that are not covered by the NVLAP accreditation.

ERA's WatR™ Supply Study, WS-93, standards were examined for any anomalies. A full review of all accuracy verification, homogeneity and stability (VHS) data was completed. All analytical verification data for all analytes in the WS-93 standards met the acceptance criteria contained in the USEPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, and the National Voluntary Laboratory Accreditation Program Handbook 150-19 for Chemical Calibration for Providers of Proficiency Testing.

The data submitted by participating laboratories was also examined for study anomalies. There were two anomalies found during the review of the study data. These anomalies are addressed on the following page.

WatR™ Supply Study, WS-93, reports shall not be reproduced except in their entirety and not without the permission of the participating laboratories. The report must not be used by the participating laboratories to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding ERA's WatR™ Supply Proficiency Testing Study, WS-93, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

- Study Discussion: WS-93 Unregulated Volatiles - Bromomethane

During the review of the WS-93 Study data ERA observed a failure rate of 62.2% for bromomethane in the Unregulated Volatiles sample. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'.

Per the requirements of ERA's NIST NVLAP PT Provider Accreditation (Lab code 200386-0) and the NELAC Standards (July 2003), ERA examined all WS-93 data sets for bimodal distribution and/or situations where results from a given method have disproportionally large failure rates. No method biases or multi-modal distributions were found in the WS-93 bromomethane data set. Per the USEPA Criteria Document, the acceptance limits for this compound are ±20% of the assigned value when the concentration is equal to or greater than 10.0 µg/L, as was the case with WS-93, and ±40% of the assigned value when the concentration is below 10.0 µg/L. Of the last forty-eight PT studies conducted by ERA, when bromomethane has been present at a concentration greater than or equal to 10 µg/L, ERA has witnessed an average failure rate of 38.4%. The average failure rate when bromomethane has been present at concentrations less than 10 µg/L is 20.6%. The USEPA conducted one study containing bromomethane at 16 µg/L, with limits of 7.98 - 22.3 µg/L or 49.9% - 139%. Obviously, the USEPA's acceptance criteria are not based on historical data from PT studies. The USEPA criteria require that this analyte be manufactured at concentrations both above and below 10.0 µg/L and randomly assigned in each study. The failure rate for bromomethane in ERA's WS-93 Study would be 17.8% using ERA's statistically determined regression equation.

The high failure rate for bromomethane causes us to question the appropriateness of the acceptance limit criteria provided in the USEPA Criteria Document. ERA will communicate this seeming disparity in failure rates to NIST/NVLAP and NELAC. When reviewing each laboratory's performance evaluation for this analyte one should take the above information into consideration.

- Study Discussion: WS-93 Unregulated Volatiles - Chloromethane

During the review of the WS-93 Study data ERA observed a failure rate of 52.3% for chloromethane in the Unregulated Volatiles sample. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'.

Per the requirements of ERA's NIST NVLAP PT Provider Accreditation (Lab code 200386-0) and the NELAC Standards (July 2003), ERA examined all WS-93 data sets for bimodal distribution and/or situations where results from a given method have disproportionally large failure rates. No method biases or multi-modal distributions were found in the WS-93 chloromethane data set. Per the USEPA Criteria Document, the acceptance limits for this compound are ±20% of the assigned value when the concentration is equal to or greater than 10.0 µg/L, as was the case with WS-93, and ±40% of the assigned value when the concentration is below 10.0 µg/L. Of the last forty-eight PT studies conducted by ERA, when chloromethane has been present at a concentration greater than or equal to 10 µg/L, ERA has witnessed an average failure rate of 39.6%. The average failure rate when chloromethane has been present at concentrations less than 10 µg/L is 14.1%. To the best of our knowledge the USEPA has never conducted a study containing chloromethane so a failure rate is not available to ERA for comparison. Obviously, the USEPA's acceptance criteria are not based on historical data from PT studies. The USEPA criteria require that this analyte be manufactured at concentrations both above and below 10.0 µg/L and randomly assigned in each study. The failure rate for chloromethane in ERA's WS-93 Study would be 18.2% using ERA's statistically determined regression equation.

The high failure rate for chloromethane causes us to question the appropriateness of the acceptance limit criteria provided in the USEPA Criteria Document. ERA will communicate this seeming disparity in failure rates to NIST/NVLAP and NELAC. When reviewing each

NVLAP® Lab code: 200386-0

laboratory's performance evaluation for this analyte one should take the above information into consideration.

ENVIRONMENTAL RESOURCE ASSOCIATES®

Study: **WS-93**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method A**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-93 Final Complete Report

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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/17/04
Study Dates: 04/12/04 - 05/27/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Metals							
1000	Aluminum	µg/L	470	463	412 - 515	Acceptable	EPA 200.7
0140	Antimony †	µg/L		38.6	27.0 - 50.2		
0001	Arsenic †	µg/L		118	103 - 132		
0002	Barium †	µg/L	1070	1130	961 - 1300	Acceptable	EPA 200.7
0141	Beryllium †	µg/L	6.09	6.73	5.72 - 7.74	Acceptable	EPA 200.7
0226	Boron †	µg/L		899	837 - 992		
0003	Cadmium †	µg/L	22.2	23.1	18.5 - 27.7	Acceptable	EPA 200.7
0004	Chromium †	µg/L	98.8	104	88.4 - 120	Acceptable	EPA 200.7
0091	Copper †	µg/L	519	536	482 - 590	Acceptable	EPA 200.7
1070	Iron	µg/L	155	164	148 - 182	Acceptable	EPA 200.7
0005	Lead †	µg/L		24.0	16.8 - 31.2		
0236	Manganese †	µg/L	106	106	97.3 - 112	Acceptable	EPA 200.7
0237	Molybdenum †	µg/L	129	142	120 - 163	Acceptable	EPA 200.7
0142	Nickel †	µg/L	97.0	97.4	82.8 - 112	Acceptable	EPA 200.7
0007	Selenium †	µg/L	96.3	92.8	74.2 - 111	Acceptable	EPA 200.7
1150	Silver	µg/L	319	317	287 - 348	Acceptable	EPA 200.7
0143	Thallium †	µg/L		4.05	2.84 - 5.27		
1185	Vanadium	µg/L	507	531	495 - 565	Acceptable	EPA 200.7
0239	Zinc †	µg/L	678	653	599 - 703	Acceptable	EPA 200.7
Mercury							
0006	Mercury †	µg/L	4.52	4.21	2.95 - 5.47	Acceptable	EPA 200.8
Inorganics							
1575	Chloride	mg/L	59.1	57.4	53.3 - 61.1	Acceptable	EPA 300.0
1610	Conductivity	µmhos/cm	502	500	473 - 527	Acceptable	EPA 120.1
0010	Fluoride †	mg/L	4.79	4.91	4.42 - 5.40	Acceptable	EPA 300.0
0009	Nitrate as N †	mg/L	4.60	4.90	4.41 - 5.39	Acceptable	EPA 300.0
1820	Nitrate + Nitrite as N	mg/L	4.60	4.90	4.41 - 5.39	Acceptable	EPA 300.0
1125	Potassium	mg/L	22.1	23.8	21.6 - 26.1	Acceptable	EPA 200.7
0145	Sulfate †	mg/L	65.0	64.5	58.0 - 70.8	Acceptable	EPA 300.0
0024	Total Dissolved Solids at 180°C †	mg/L	394	331	215 - 447	Acceptable	EPA 160.1
Nitrite							
0092	Nitrite as N †	mg/L	1.81	1.66	1.41 - 1.91	Acceptable	EPA 300.0
Total Organic Carbon							
0263	Total organic carbon †	mg/L	3.77	2.02	1.73 - 2.45	Not Acceptable	EPA 415.1

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-93 Final Complete Report

Tenkasi Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/17/04
Study Dates: 04/12/04 - 05/27/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Volatiles							
0039	Benzene †	µg/L	15.9	15.8	12.6 - 19.0	Acceptable	EPA 524.2
0037	Carbon tetrachloride †	µg/L	11.3	12.8	10.2 - 15.4	Acceptable	EPA 524.2
0049	Chlorobenzene †	µg/L	16.1	15.6	12.5 - 18.7	Acceptable	EPA 524.2
0054	1,2-Dichlorobenzene †	µg/L	9.24	8.97	5.38 - 12.6	Acceptable	EPA 524.2
0041	1,4-Dichlorobenzene †	µg/L	8.39	7.71	4.63 - 10.8	Acceptable	EPA 524.2
0035	1,2-Dichloroethane †	µg/L	9.84	9.67	5.80 - 13.5	Acceptable	EPA 524.2
0034	1,1-Dichloroethylene †	µg/L	18.4	18.7	15.0 - 22.4	Acceptable	EPA 524.2
0043	cis-1,2-Dichloroethylene †	µg/L	7.76	7.56	4.54 - 10.6	Acceptable	EPA 524.2
0042	trans-1,2-Dichloroethylene †	µg/L	9.10	9.08	5.45 - 12.7	Acceptable	EPA 524.2
0044	1,2-Dichloropropane †	µg/L	3.17	3.12	1.87 - 4.37	Acceptable	EPA 524.2
0048	Ethylbenzene †	µg/L	7.10	7.61	4.57 - 10.7	Acceptable	EPA 524.2
0055	Methylene chloride †	µg/L	9.97	10.1	8.08 - 12.1	Acceptable	EPA 524.2
0053	Styrene †	µg/L	9.18	9.50	5.70 - 13.3	Acceptable	EPA 524.2
0040	Tetrachloroethylene †	µg/L	5.36	6.15	3.69 - 8.61	Acceptable	EPA 524.2
0047	Toluene †	µg/L	8.73	8.77	5.26 - 12.3	Acceptable	EPA 524.2
0076	1,2,4-Trichlorobenzene †	µg/L	5.89	6.13	3.68 - 8.58	Acceptable	EPA 524.2
0036	1,1,1-Trichloroethane †	µg/L	4.71	4.86	2.92 - 6.80	Acceptable	EPA 524.2
0061	1,1,2-Trichloroethane †	µg/L	5.03	4.86	2.92 - 6.80	Acceptable	EPA 524.2
0038	Trichloroethylene †	µg/L	4.27	4.93	2.96 - 6.90	Acceptable	EPA 524.2
0032	Vinyl chloride †	µg/L	8.25	6.80	4.08 - 9.52	Acceptable	EPA 524.2
0090	Xylenes, total †	µg/L	31.6	33.1	26.5 - 39.7	Acceptable	EPA 524.2
Halomethanes							
0019	Bromodichloromethane †	µg/L	12.4	12.6	10.1 - 15.1	Acceptable	EPA 524.2
0018	Bromoform †	µg/L	21.0	21.6	17.3 - 25.9	Acceptable	EPA 524.2
0020	Chlorodibromomethane †	µg/L	15.4	14.8	11.8 - 17.8	Acceptable	EPA 524.2
0017	Chloroform †	µg/L	32.5	34.1	27.3 - 40.9	Acceptable	EPA 524.2
0021	Total Trihalomethanes †	µg/L	81.2	83.1	66.5 - 99.7	Acceptable	EPA 524.2
Unregulated Volatiles							
0067	Bromobenzene †	µg/L	5.26	5.75	3.45 - 8.05	Acceptable	EPA 524.2
0089	Bromochloromethane †	µg/L	6.85	7.29	4.37 - 10.2	Acceptable	EPA 524.2
0069	Bromomethane †	µg/L	9.27	10.7	8.56 - 12.8	Acceptable	EPA 524.2
0079	n-Butylbenzene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0086	sec-Butylbenzene †	µg/L	7.61	7.47	4.48 - 10.5	Acceptable	EPA 524.2
0085	tert-Butylbenzene †	µg/L	19.2	19.0	15.2 - 22.8	Acceptable	EPA 524.2
5000	tert-Butylmethyl ether (MTBE)	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0070	Chloroethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0068	Chloromethane †	µg/L	23.5	14.0	11.2 - 16.8	Not Acceptable	EPA 524.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
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NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-93 Final Complete Report

Tenkasi Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/17/04
Study Dates: 04/12/04 - 05/27/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Unregulated Volatiles (Cont.)							
0071	2-Chlorotoluene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0072	4-Chlorotoluene †	µg/L	7.33	7.06	4.24 - 9.88	Acceptable	EPA 524.2
0057	Dibromomethane †	µg/L	4.96	4.94	2.96 - 6.92	Acceptable	EPA 524.2
0066	1,3-Dichlorobenzene †	µg/L	9.90	9.19	5.51 - 12.9	Acceptable	EPA 524.2
0088	Dichlorodifluoromethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0056	1,1-Dichloroethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0059	1,3-Dichloropropane †	µg/L	9.71	9.87	5.92 - 13.8	Acceptable	EPA 524.2
0060	2,2-Dichloropropane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0058	1,1-Dichloropropene †	µg/L	4.99	5.43	3.26 - 7.60	Acceptable	EPA 524.2
0152	cis-1,3-Dichloropropene †	µg/L	5.72	6.50	3.90 - 9.10	Acceptable	EPA 524.2
0153	trans-1,3-Dichloropropene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0087	Fluorotrichloromethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0081	Hexachlorobutadiene †	µg/L	8.30	8.24	4.94 - 11.5	Acceptable	EPA 524.2
0084	Isopropylbenzene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0083	4-Isopropyltoluene †	µg/L	5.95	5.80	3.48 - 8.12	Acceptable	EPA 524.2
0078	n-Propylbenzene †	µg/L	5.07	5.35	3.21 - 7.49	Acceptable	EPA 524.2
0063	1,1,1,2-Tetrachloroethane †	µg/L	7.70	9.60	5.76 - 13.4	Acceptable	EPA 524.2
0065	1,1,2,2-Tetrachloroethane †	µg/L	3.75	3.48	2.09 - 4.87	Acceptable	EPA 524.2
0077	1,2,3-Trichlorobenzene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0064	1,2,3-Trichloropropane †	µg/L	20.5	18.3	14.6 - 22.0	Acceptable	EPA 524.2
0075	1,2,4-Trimethylbenzene †	µg/L	9.64	8.87	5.32 - 12.4	Acceptable	EPA 524.2
0082	1,3,5-Trimethylbenzene †	µg/L	17.5	18.0	14.4 - 21.6	Acceptable	EPA 524.2
Regulated Pesticides							
0093	Alachlor †	µg/L	< 0.2	3.12	1.72 - 4.52	Not Acceptable	EPA 525.2
0256	Aldrin †	µg/L	0.371	0.561	0.226 - 0.707	Acceptable	EPA 508.1
0094	Atrazine †	µg/L	< 0.2	12.4	6.82 - 18.0	Not Acceptable	EPA 525.2
7160	Butachlor	µg/L		0.811	0.395 - 1.12		
7410	Diazinon	µg/L		0.00			
0258	Dieldrin †	µg/L	1.29	1.86	1.30 - 2.33	Not Acceptable	EPA 508.1
0011	Endrin †	µg/L	2.84	3.29	2.30 - 4.28	Acceptable	EPA 508.1
0095	Heptachlor †	µg/L	2.40	2.93	1.61 - 4.25	Acceptable	EPA 508.1
0096	Heptachlor epoxide (beta) †	µg/L	3.50	4.20	2.31 - 6.09	Acceptable	EPA 508.1
0172	Hexachlorobenzene †	µg/L	< 0.2	0.768	0.344 - 1.02	Not Acceptable	EPA 525.2
0112	Hexachlorocyclopentadiene †	µg/L	< 0.2	3.53	0.119 - 4.51	Not Acceptable	EPA 525.2
0012	gamma-BHC (Lindane) †	µg/L	0.580	0.815	0.448 - 1.18	Acceptable	EPA 508.1
0013	Methoxychlor †	µg/L	43.1	53.7	29.5 - 77.9	Acceptable	EPA 508.1
7835	Metolachlor	µg/L		0.00			
7845	Metribuzin	µg/L		24.9	10.6 - 32.0		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-93 Final Complete Report

Tenkasi Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/17/04
Study Dates: 04/12/04 - 05/27/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Pesticides (Cont.)							
7875	Molinate (ordram)	µg/L		12.3	6.10 - 17.5		
8035	Prometon	µg/L		0.00			
0259	Propachlor †	µg/L		1.44	0.779 - 2.15		
0113	Simazine †	µg/L	< 0.2	14.0	3.22 - 20.6	Not Acceptable	EPA 525.2
8220	Thiobencarb	µg/L		0.00			
0244	Trifluralin †	µg/L		4.71	2.16 - 6.37		
Toxaphene							
0014	Toxaphene †	µg/L	3.11	3.19	1.75 - 4.63	Acceptable	EPA 508.1
Chlordane							
0097	Chlordane, technical †	µg/L	15.0	16.8	9.24 - 24.4	Acceptable	EPA 508.1
EDB/DBCP							
0045	Dibromochloropropane (DBCP) †	µg/L	0.877	0.604	0.362 - 0.846	Not Acceptable	EPA 504.1
0046	Ethylene Dibromide (EDB) †	µg/L	1.31	1.01	0.606 - 1.41	Acceptable	EPA 504.1
5180	1,2,3-Trichloropropane	µg/L	1.59	1.68	0.931 - 2.22	Acceptable	EPA 504.1
Regulated Semivolatiles #1							
5500	Acenaphthene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5505	Acenaphthylene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5555	Anthracene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5575	Benzo(a)anthracene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5585	Benzo(b)fluoranthene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5600	Benzo(k)fluoranthene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5590	Benzo(g,h,i)perylene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
0122	Benzo(a)pyrene †	µg/L	0.31	0.379	0.113 - 0.525	Acceptable	EPA 525.2
5670	Butylbenzylphthalate	µg/L	11.5	10.9	4.02 - 16.5	Acceptable	EPA 525.2
5855	Chrysene	µg/L	0.72	1.01	0.569 - 1.34	Acceptable	EPA 525.2
5895	Dibenz(a,h)anthracene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5925	Di-n-butylphthalate	µg/L	42.3	39.0	16.5 - 59.3	Acceptable	EPA 525.2
6070	Diethylphthalate	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
6135	Dimethylphthalate	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
6200	Di-n-octylphthalate	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
0134	bis(2-Ethylhexyl)adipate †	µg/L	11.7	30.4	12.1 - 44.0	Not Acceptable	EPA 525.2
0136	bis(2-Ethylhexyl)phthalate †	µg/L	20.2	20.7	8.51 - 32.1	Acceptable	EPA 525.2
6265	Fluoranthene	µg/L	0.97	1.38	0.424 - 1.92	Acceptable	EPA 525.2
6270	Fluorene	µg/L	5.63	8.52	4.17 - 11.1	Acceptable	EPA 525.2
6315	Indeno(1,2,3-cd)pyrene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
5005	Naphthalene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2

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NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-93 Final Complete Report

Tenkasi Viswanathan
QA Director
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Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/17/04
Study Dates: 04/12/04 - 05/27/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Semivolatiles #1 (Cont.)							
6615	Phenanthrene	µg/L	1.80	2.48	1.42 - 3.20	Acceptable	EPA 525.2
6665	Pyrene	µg/L	< 0.2	0.00		Acceptable	EPA 525.2
Regulated Herbicides							
0262	Acifluorfen †	µg/L	32.0	46.4	22.1 - 60.5	Acceptable	EPA 515.4
8530	Bentazon	µg/L		120	57.8 - 168		
8540	Chloramben	µg/L		0.00			
0015	2,4-D †	µg/L		61.2	30.6 - 91.8		
8560	2,4-DB	µg/L	128	131	33.4 - 181	Acceptable	EPA 515.4
8550	Dacthal diacid (DCPA)	µg/L		0.00			
0115	Dalapon †	µg/L	1.44	137	0.00 - 185	Acceptable	EPA 515.4
0247	Dicamba †	µg/L		10.4	3.33 - 15.3		
8600	3,5-Dichlorobenzoic acid	µg/L	< 1	0.00		Acceptable	EPA 515.4
8605	Dichlorprop	µg/L	84.4	127	34.2 - 172	Acceptable	EPA 515.4
0116	Dinoseb †	µg/L	25.3	22.1	0.317 - 33.1	Acceptable	EPA 515.4
6500	4-Nitrophenol	µg/L	< 1	0.00		Acceptable	EPA 515.4
0102	Pentachlorophenol †	µg/L	32.4	25.7	12.9 - 38.6	Acceptable	EPA 515.4
0117	Picloram †	µg/L	8.82	64.7	0.00 - 85.7	Acceptable	EPA 515.4
8655	2,4,5-T	µg/L	37.6	59.4	24.9 - 90.1	Acceptable	EPA 515.4
0016	2,4,5-TP (Silvex) †	µg/L	77.4	89.7	44.9 - 135	Acceptable	EPA 515.4

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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/17/04
Study Dates: 04/12/04 - 05/27/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Perchlorate							
1895	Perchlorate	µg/L	8.63	9.11	7.32 - 10.1	Acceptable	EPA 314
WS PCBs in Water							
8912	Aroclor 1016/1242	µg/L	< 0.1	0.00		Acceptable	EPA 508.1
8890	Aroclor 1232	µg/L	< 0.1	0.00		Acceptable	EPA 508.1
8900	Aroclor 1248	µg/L	2.27	1.91	1.08 - 2.30	Acceptable	EPA 508.1
8905	Aroclor 1254	µg/L	< 0.1	0.00		Acceptable	EPA 508.1
8910	Aroclor 1260	µg/L	< 0.1	0.00		Acceptable	EPA 508.1

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NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

Study: **WS-93**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method B**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-93 Final Complete Report

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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/17/04
Study Dates: 04/12/04 - 05/27/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Metals							
1000	Aluminum	µg/L	442	463	412 - 515	Acceptable	EPA 200.8
0140	Antimony †	µg/L	38.3	38.6	27.0 - 50.2	Acceptable	EPA 200.8
0001	Arsenic †	µg/L	111	118	103 - 132	Acceptable	EPA 200.8
0002	Barium †	µg/L	1136	1130	961 - 1300	Acceptable	EPA 200.8
0141	Beryllium †	µg/L	6.39	6.73	5.72 - 7.74	Acceptable	EPA 200.8
0226	Boron †	µg/L	866	899	837 - 992	Acceptable	EPA 200.8
0003	Cadmium †	µg/L	21.4	23.1	18.5 - 27.7	Acceptable	EPA 200.8
0004	Chromium †	µg/L	105	104	88.4 - 120	Acceptable	EPA 200.8
0091	Copper †	µg/L	573	536	482 - 590	Acceptable	EPA 200.8
1070	Iron	µg/L	161	164	148 - 182	Acceptable	EPA 200.8
0005	Lead †	µg/L	23.0	24.0	16.8 - 31.2	Acceptable	EPA 200.8
0236	Manganese †	µg/L	107	106	97.3 - 112	Acceptable	EPA 200.8
0237	Molybdenum †	µg/L	139	142	120 - 163	Acceptable	EPA 200.8
0142	Nickel †	µg/L	96.2	97.4	82.8 - 112	Acceptable	EPA 200.8
0007	Selenium †	µg/L	93.7	92.8	74.2 - 111	Acceptable	EPA 200.8
1150	Silver	µg/L	286	317	287 - 348	Not Acceptable	EPA 200.8
0143	Thallium †	µg/L	3.86	4.05	2.84 - 5.27	Acceptable	EPA 200.8
1185	Vanadium	µg/L	527	531	495 - 565	Acceptable	EPA 200.8
0239	Zinc †	µg/L	651	653	599 - 703	Acceptable	EPA 200.8
Mercury							
0006	Mercury †	µg/L	4.77	4.21	2.95 - 5.47	Acceptable	EPA 245.1
Inorganics							
1575	Chloride	mg/L	56.7	57.4	53.3 - 61.1	Acceptable	EPA 325.3
1610	Conductivity	µmhos/cm		500	473 - 527		
0010	Fluoride †	mg/L		4.91	4.42 - 5.40		
0009	Nitrate as N †	mg/L	4.88	4.90	4.41 - 5.39	Acceptable	EPA 353.2
1820	Nitrate + Nitrite as N	mg/L	4.88	4.90	4.41 - 5.39	Acceptable	EPA 353.2
1125	Potassium	mg/L		23.8	21.6 - 26.1		
0145	Sulfate †	mg/L	59.5	64.5	58.0 - 70.8	Acceptable	EPA 375.4
0024	Total Dissolved Solids at 180°C †	mg/L		331	215 - 447		
Nitrite							
0092	Nitrite as N †	mg/L	1.63	1.66	1.41 - 1.91	Acceptable	EPA 353.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
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NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-99 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 EPA ID: KS00902

Report Issued: 12/15/04
Study Dates: 10/08/04 - 11/22/04

WS Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Values are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, and the National Environmental Laboratory Accreditation Conference (NELAC) program criteria as applicable. A parameter not added to the standard is given an Assigned Value of "0" per the guidelines contained in the USEPA's Criteria Document and NELAC standards.

The Acceptance Limits are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, the NELAC PT program criteria or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside of the Acceptance Limits.
No Evaluation	=	Reported Value cannot be evaluated.

The Method Description is the method the laboratory reported to ERA.

WS Study Discussion:

ERA's WatR™ Supply Proficiency Testing Study, WS-99, has been reviewed by ERA Senior Management and certified compliant with the requirements of the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document (December 1998), the NELAC Standards Chapter 2 and Associated Appendices, applicable state PT programs, and those contained in the National Institute of Standards and Technology NVLAP Handbooks 150 and 150-19. ERA is a NIST NVLAP accredited PT Provider (Lab Code 200386-0).

This report contains data that are not covered by the NVLAP accreditation.

ERA's WatR™ Supply Study, WS-99, standards were examined for any anomalies. A full review of all accuracy verification, homogeneity and stability (VHS) data was completed. All analytical verification data for all analytes in the WS-99 standards met the acceptance criteria contained in the USEPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, and the National Voluntary Laboratory Accreditation Program Handbook 150-19 for Chemical Calibration for Providers of Proficiency Testing.

The data submitted by participating laboratories was also examined for study anomalies. There was one anomaly found during the review of the study data. This anomaly is addressed on the following page.

WatR™ Supply Study, WS-99, reports shall not be reproduced except in their entirety and not without the permission of the participating laboratories. The report must not be used by the participating laboratories to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding ERA's WatR™ Supply Proficiency Testing Study, WS-99, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

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- Study Discussion: WS-99 Unregulated Volatiles - Dichlorodifluoromethane

During the review of the WS-99 Study data ERA observed a failure rate of 69.4% for dichlorodifluoromethane in the Unregulated Volatiles sample. As we believe this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'. Per the requirements of ERA's NIST NVLAP PT Provider Accreditation (Lab code 200386-0) and the NELAC Standards (July 2003), ERA examined all WS-99 data sets for bimodal distribution and/or situations where results from a given method have disproportionately large failure rates. No method biases or multi-modal distributions were found in the WS-99 dichlorodifluoromethane data set.

Per the USEPA Criteria Document, the acceptance limits for this compound are ±20% of the assigned value when the concentration is greater than or equal to 10.0 µg/L, as was the case with WS-99, and ±40% of the assigned value when the concentration is less than 10.0 µg/L. Of the last forty-eight PT studies conducted by ERA, when dichlorodifluoromethane has been present at a concentration greater than or equal to 10 µg/L, ERA has witnessed an average failure rate of 56.7%. The average failure rate when dichlorodifluoromethane has been present at concentrations less than 10 µg/L is 30.6%. To the best of our knowledge the USEPA never conducted a study containing dichlorodifluoromethane so a failure rate is not available to ERA for comparison. The USEPA criteria require that this analyte be manufactured at concentrations both above and below 10.0 µg/L and randomly assigned in each study. The failure rate for dichlorodifluoromethane in ERA's WS-99 Study would be 19.4% using ERA's statistically determined regression equation.

The high failure rate causes us to question the appropriateness of the acceptance limit criteria provided in the USEPA Criteria Document. ERA will communicate this seeming disparity in failure rates to NIST/NVLAP. When reviewing each laboratory's performance evaluation for this analyte one should take the above information into consideration.

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Study: **WS-99**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method A**

ACCREDITED
Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP
Lab Code: 200386-0

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-99 Final Complete Report

Tenkasi Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/15/04
Study Dates: 10/08/04 - 11/22/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Metals							
1000	Aluminum	µg/L	707	719	644 - 792	Acceptable	EPA 200.7
0140	Antimony †	µg/L		18.8	13.2 - 24.4		
0001	Arsenic †	µg/L	106	102	89.3 - 114	Acceptable	EPA 200.7
0002	Barium †	µg/L	2360	2450	2080 - 2820	Acceptable	EPA 200.7
0141	Beryllium †	µg/L	6.88	6.61	5.62 - 7.60	Acceptable	EPA 200.7
0226	Boron †	µg/L		541	508 - 592		
0003	Cadmium †	µg/L	11.8	12.1	9.68 - 14.5	Acceptable	EPA 200.7
0004	Chromium †	µg/L	86.6	83.8	71.2 - 96.4	Acceptable	EPA 200.7
0091	Copper †	µg/L	408	407	366 - 448	Acceptable	EPA 200.7
1070	Iron	µg/L	667	648	595 - 704	Acceptable	EPA 200.7
0005	Lead †	µg/L		66.4	46.5 - 86.3		
0236	Manganese †	µg/L	487	471	438 - 495	Acceptable	EPA 200.7
0237	Molybdenum †	µg/L	40.1	42.7	35.6 - 49.6	Acceptable	EPA 200.7
0142	Nickel †	µg/L	153	152	129 - 175	Acceptable	EPA 200.7
0007	Selenium †	µg/L		34.1	27.3 - 40.9		
1150	Silver	µg/L	495	488	442 - 536	Acceptable	EPA 200.7
0143	Thallium †	µg/L		8.75	6.13 - 11.4		
1185	Vanadium	µg/L	463	467	435 - 497	Acceptable	EPA 200.7
0239	Zinc †	µg/L	1020	959	881 - 1030	Acceptable	EPA 200.7
Mercury							
0006	Mercury †	µg/L	6.85	7.38	5.17 - 9.59	Acceptable	EPA 200.8
Inorganics							
1575	Chloride	mg/L	60.3	57.6	53.5 - 61.3	Acceptable	EPA 325.3
1610	Conductivity	µmhos/cm	506	510	483 - 537	Acceptable	EPA 120.1
0010	Fluoride †	mg/L		5.56	5.00 - 6.12		
0009	Nitrate as N †	mg/L	3.34	3.56	3.20 - 3.92	Acceptable	EPA 353.2
1820	Nitrate + Nitrite as N	mg/L	3.34	3.56	3.20 - 3.92	Acceptable	EPA 353.2
1125	Potassium	mg/L	24.6	21.4	19.4 - 23.5	Not Acceptable	EPA 200.7
0145	Sulfate †	mg/L	23.5	21.4	18.7 - 23.8	Acceptable	EPA 375.4
0024	Total Dissolved Solids at 180°C †	mg/L	364	421	270 - 572	Acceptable	EPA 160.1
Nitrite							
0092	Nitrite as N †	mg/L	1.99	1.91	1.62 - 2.20	Acceptable	EPA 353.2
Total Organic Carbon							
0263	Total organic carbon †	mg/L	2.25	2.30	1.99 - 2.75	Acceptable	EPA 415.1

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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WS-99 Final Complete Report

Tenkasi Viswanathan
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15130 S Keeler
Suite B
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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/15/04
Study Dates: 10/08/04 - 11/22/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Volatiles							
0039	Benzene †	µg/L	8.91	9.12	5.47 - 12.8	Acceptable	EPA 524.2
0037	Carbon tetrachloride †	µg/L	8.07	8.81	5.29 - 12.3	Acceptable	EPA 524.2
0049	Chlorobenzene †	µg/L	63.4	47.6	38.1 - 57.1	Not Acceptable	EPA 524.2
0054	1,2-Dichlorobenzene †	µg/L	14.9	10.5	8.40 - 12.6	Not Acceptable	EPA 524.2
0041	1,4-Dichlorobenzene †	µg/L	14.3	11.3	9.04 - 13.6	Not Acceptable	EPA 524.2
0035	1,2-Dichloroethane †	µg/L	4.67	4.52	2.71 - 6.33	Acceptable	EPA 524.2
0034	1,1-Dichloroethylene †	µg/L	4.43	4.19	2.51 - 5.87	Acceptable	EPA 524.2
0043	cis-1,2-Dichloroethylene †	µg/L	18.6	18.0	14.4 - 21.6	Acceptable	EPA 524.2
0042	trans-1,2-Dichloroethylene †	µg/L	24.6	24.6	19.7 - 29.5	Acceptable	EPA 524.2
0044	1,2-Dichloropropane †	µg/L	8.90	8.02	4.81 - 11.2	Acceptable	EPA 524.2
0048	Ethylbenzene †	µg/L	15.2	12.5	10.0 - 15.0	Not Acceptable	EPA 524.2
0055	Methylene chloride †	µg/L	19.1	19.2	15.4 - 23.0	Acceptable	EPA 524.2
0053	Styrene †	µg/L	6.66	4.42	2.65 - 6.19	Not Acceptable	EPA 524.2
0040	Tetrachloroethylene †	µg/L	4.25	2.54	1.52 - 3.56	Not Acceptable	EPA 524.2
0047	Toluene †	µg/L	10.4	8.12	4.87 - 11.4	Acceptable	EPA 524.2
0076	1,2,4-Trichlorobenzene †	µg/L	25.6	11.8	9.44 - 14.2	Not Acceptable	EPA 524.2
0036	1,1,1-Trichloroethane †	µg/L	19.1	18.8	15.0 - 22.6	Acceptable	EPA 524.2
0061	1,1,2-Trichloroethane †	µg/L	17.8	11.6	9.28 - 13.9	Not Acceptable	EPA 524.2
0038	Trichloroethylene †	µg/L	14.3	9.20	5.52 - 12.9	Not Acceptable	EPA 524.2
0032	Vinyl chloride †	µg/L	12.5	11.4	6.84 - 16.0	Acceptable	EPA 524.2
0090	Xylenes, total †	µg/L	54.5	46.6	37.3 - 55.9	Acceptable	EPA 524.2
Halomethanes							
0019	Bromodichloromethane †	µg/L	12.3	12.6	10.1 - 15.1	Acceptable	EPA 524.2
0018	Bromoform †	µg/L	36.7	35.5	28.4 - 42.6	Acceptable	EPA 524.2
0020	Chlorodibromomethane †	µg/L	49.1	47.8	38.2 - 57.4	Acceptable	EPA 524.2
0017	Chloroform †	µg/L	12.0	13.6	10.9 - 16.3	Acceptable	EPA 524.2
0021	Total Trihalomethanes †	µg/L	110	110	88.0 - 132	Acceptable	EPA 524.2
Unregulated Volatiles							
0067	Bromobenzene †	µg/L	6.55	6.39	3.83 - 8.95	Acceptable	EPA 524.2
0089	Bromochloromethane †	µg/L	17.0	16.5	13.2 - 19.8	Acceptable	EPA 524.2
0069	Bromomethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0079	n-Butylbenzene †	µg/L	6.55	0.00		Not Acceptable	EPA 524.2
0086	sec-Butylbenzene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0085	tert-Butylbenzene †	µg/L	3.86	2.74	1.64 - 3.84	Not Acceptable	EPA 524.2
5000	tert-Butylmethyl ether (MTBE)	µg/L	22.6	26.7	19.5 - 34.7	Acceptable	EPA 524.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-99 Final Complete Report

Tenkasi Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/15/04
Study Dates: 10/08/04 - 11/22/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Unregulated Volatiles (Cont.)							
0070	Chloroethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0068	Chloromethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0071	2-Chlorotoluene †	µg/L	16.2	18.9	15.1 - 22.7	Acceptable	EPA 524.2
0072	4-Chlorotoluene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0057	Dibromomethane †	µg/L	10.6	7.11	4.27 - 9.95	Not Acceptable	EPA 524.2
0066	1,3-Dichlorobenzene †	µg/L	10.6	9.06	5.44 - 12.7	Acceptable	EPA 524.2
0088	Dichlorodifluoromethane †	µg/L	23.8	18.0	14.4 - 21.6	Not Acceptable	EPA 524.2
0056	1,1-Dichloroethane †	µg/L	15.3	16.2	13.0 - 19.4	Acceptable	EPA 524.2
0059	1,3-Dichloropropane †	µg/L	11.0	7.35	4.41 - 10.3	Not Acceptable	EPA 524.2
0060	2,2-Dichloropropane †	µg/L	12.0	14.6	11.7 - 17.5	Acceptable	EPA 524.2
0058	1,1-Dichloropropene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0152	cis-1,3-Dichloropropene †	µg/L	16.1	15.6	12.5 - 18.7	Acceptable	EPA 524.2
0153	trans-1,3-Dichloropropene †	µg/L	3.14	2.91	1.75 - 4.07	Acceptable	EPA 524.2
0087	Fluorotrichloromethane †	µg/L	5.96	9.06	5.44 - 12.7	Acceptable	EPA 524.2
0081	Hexachlorobutadiene †	µg/L	31.8	14.5	11.6 - 17.4	Not Acceptable	EPA 524.2
0084	Isopropylbenzene †	µg/L	20.0	17.1	13.7 - 20.5	Acceptable	EPA 524.2
0083	4-Isopropyltoluene †	µg/L	13.5	11.6	9.28 - 13.9	Acceptable	EPA 524.2
0078	n-Propylbenzene †	µg/L	8.23	9.53	5.72 - 13.3	Acceptable	EPA 524.2
0063	1,1,1,2-Tetrachloroethane †	µg/L	10.7	8.46	5.08 - 11.8	Acceptable	EPA 524.2
0065	1,1,2,2-Tetrachloroethane †	µg/L	11.0	10.2	8.16 - 12.2	Acceptable	EPA 524.2
0077	1,2,3-Trichlorobenzene †	µg/L	24.6	9.35	5.61 - 13.1	Not Acceptable	EPA 524.2
0064	1,2,3-Trichloropropane †	µg/L	12.8	10.6	8.48 - 12.7	Not Acceptable	EPA 524.2
0075	1,2,4-Trimethylbenzene †	µg/L	10.5	10.3	8.24 - 12.4	Acceptable	EPA 524.2
0082	1,3,5-Trimethylbenzene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
Regulated Pesticides							
0093	Alachlor †	µg/L		2.99	1.64 - 4.34		
0256	Aldrin †	µg/L	1.03	1.94	0.826 - 2.44	Acceptable	EPA 508
0094	Atrazine †	µg/L		5.58	3.07 - 8.09		
7160	Butachlor	µg/L		0.00			
7410	Diazinon	µg/L		0.00			
0258	Dieldrin †	µg/L	0.475	0.743	0.521 - 0.933	Not Acceptable	EPA 508
0011	Endrin †	µg/L	2.65	3.95	2.77 - 5.14	Not Acceptable	EPA 508
0095	Heptachlor †	µg/L	1.27	2.75	1.51 - 3.99	Not Acceptable	EPA 508
0096	Heptachlor epoxide (beta) †	µg/L	2.95	4.73	2.60 - 6.86	Acceptable	EPA 508
0172	Hexachlorobenzene †	µg/L		1.08	0.489 - 1.41		
0112	Hexachlorocyclopentadiene †	µg/L		4.58	0.112 - 5.84		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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WS-99 Final Complete Report

Tenkasi Viswanathan
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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/15/04
Study Dates: 10/08/04 - 11/22/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Pesticides (Cont.)							
0012	gamma-BHC (Lindane) †	µg/L	1.31	3.12	1.72 - 4.52	Not Acceptable	EPA 508
0013	Methoxychlor †	µg/L	8.34	14.6	8.03 - 21.2	Acceptable	EPA 508
7835	Metolachlor	µg/L		0.00			
7845	Metribuzin	µg/L		73.0	14.7 - 112		
7875	Molinate (ordram)	µg/L		67.6	33.5 - 96.0		
8035	Prometon	µg/L		66.2	41.9 - 66.2		
0259	Propachlor †	µg/L		1.91	1.10 - 2.75		
0113	Simazine †	µg/L		6.41	1.59 - 10.3		
8220	Thiobencarb	µg/L		0.00			
0244	Trifluralin †	µg/L		1.56	0.598 - 1.89		
Toxaphene							
0014	Toxaphene †	µg/L	13.7	14.5	7.98 - 21.0	Acceptable	EPA 508
Chlordane							
0097	Chlordane, technical †	µg/L	10.2	14.3	7.87 - 20.7	Acceptable	EPA 508
EDB/DBCP							
0045	Dibromochloropropane (DBCP) †	µg/L	2.07	1.97	1.18 - 2.76	Acceptable	EPA 504.1
0046	Ethylene Dibromide (EDB) †	µg/L	0.238	0.231	0.139 - 0.323	Acceptable	EPA 504.1
5180	1,2,3-Trichloropropane	µg/L	0.165	0.149	0.0825 - 0.197	Acceptable	EPA 504.1
Regulated Semivolatiles #1							
5500	Acenaphthene	µg/L	< 0.1	0.00		Acceptable	EPA 525.2
5505	Acenaphthylene	µg/L	< 0.1	0.00		Acceptable	EPA 525.2
5555	Anthracene	µg/L	< 0.1	0.00		Acceptable	EPA 525.2
5575	Benzo(a)anthracene	µg/L	1.69	1.94	0.871 - 2.50	Acceptable	EPA 525.2
5585	Benzo(b)fluoranthene	µg/L	< 0.1	0.00		Acceptable	EPA 525.2
5600	Benzo(k)fluoranthene	µg/L	< 0.1	0.00		Acceptable	EPA 525.2
5590	Benzo(g,h,i)perylene	µg/L	0.81	1.92	0.434 - 3.01	Acceptable	EPA 525.2
0122	Benzo(a)pyrene †	µg/L	0.72	1.05	0.260 - 1.37	Acceptable	EPA 525.2
5670	Butylbenzylphthalate	µg/L	< 2.0	0.00		Acceptable	EPA 525.2
5855	Chrysene	µg/L	< 0.1	0.00		Acceptable	EPA 525.2
5895	Dibenz(a,h)anthracene	µg/L	< 0.1	0.00		Acceptable	EPA 525.2
5925	Di-n-butylphthalate	µg/L	41.7	48.1	20.4 - 73.1	Acceptable	EPA 525.2
6070	Diethylphthalate	µg/L		0.00			
6135	Dimethylphthalate	µg/L	< 2.0	0.00		Acceptable	EPA 525.2
6200	Di-n-octylphthalate	µg/L	32.6	43.8	22.4 - 57.4	Acceptable	EPA 525.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Semivolatiles #1 (Cont.)							
0134	bis(2-Ethylhexyl)adipate †	µg/L	< 1.6	47.6	20.2 - 68.4	Not Acceptable	EPA 525.2
0136	bis(2-Ethylhexyl)phthalate †	µg/L	24.1	32.9	14.0 - 51.2	Acceptable	EPA 525.2
6265	Fluoranthene	µg/L	0.34	0.423	0.118 - 0.613	Acceptable	EPA 525.2
6270	Fluorene	µg/L	< 0.1	0.00		Acceptable	EPA 525.2
6315	Indeno(1,2,3-cd)pyrene	µg/L	< 0.1	0.00		Acceptable	EPA 525.2
5005	Naphthalene	µg/L	2.32	2.82	1.08 - 3.69	Acceptable	EPA 525.2
6615	Phenanthrene	µg/L	< 0.1	0.00		Acceptable	EPA 525.2
6665	Pyrene	µg/L	< 0.1	0.00		Acceptable	EPA 525.2
Regulated Herbicides							
0262	Acifluorfen †	µg/L	25.6	15.5	0.148 - 27.6	Acceptable	EPA 515.4
8530	Bentazon	µg/L	21.3	140	67.5 - 196	Not Acceptable	EPA 515.4
8540	Chloramben	µg/L		0.00			
0015	2,4-D †	µg/L	50.7	146	73.0 - 219	Not Acceptable	EPA 515.4
8560	2,4-DB	µg/L	5.75	3.63	0.00 - 17.6	Acceptable	EPA 515.4
8550	Dacthal diacid (DCPA)	µg/L		0.00			
0115	Dalapon †	µg/L	< 10	142	0.00 - 192	Acceptable	EPA 515.4
0247	Dicamba †	µg/L	5.5	93.5	24.4 - 129	Not Acceptable	EPA 515.4
8600	3,5-Dichlorobenzoic acid	µg/L	< 1	0.00		Acceptable	EPA 515.4
8605	Dichlorprop	µg/L	< 1	0.00		Acceptable	EPA 515.4
0116	Dinoseb †	µg/L	47.1	48.6	2.13 - 73.2	Acceptable	EPA 515.4
6500	4-Nitrophenol	µg/L	91.2	142	0.00 - 244	Acceptable	EPA 515.4
0102	Pentachlorophenol †	µg/L	7.75	7.53	3.77 - 11.3	Acceptable	EPA 515.4
0117	Picloram †	µg/L	< 1	20.3	0.489 - 29.4	Not Acceptable	EPA 515.4
8655	2,4,5-T	µg/L		146	36.5 - 218		
0016	2,4,5-TP (Silvex) †	µg/L	9.84	6.35	3.18 - 9.53	Not Acceptable	EPA 515.4

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ERA Laboratory Code: A5230-01
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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Perchlorate							
1895	Perchlorate	µg/L	5.34	5.14	4.13 - 5.71	Acceptable	EPA 314
WS PCBs in Water							
8912	Aroclor 1016/1242	µg/L	< 0.5	0.00		Acceptable	EPA 508
8890	Aroclor 1232	µg/L	< 0.5	0.00		Acceptable	EPA 508
8900	Aroclor 1248	µg/L	< 0.5	0.00		Acceptable	EPA 508
8905	Aroclor 1254	µg/L	< 0.5	0.00		Acceptable	EPA 508
8910	Aroclor 1260	µg/L	1.94	2.31	1.40 - 2.74	Acceptable	EPA 508

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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Study: **WS-99**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method B**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-99 Final Complete Report

Tenkasi Viswanathan
QA Director
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15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/15/04
Study Dates: 10/08/04 - 11/22/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Metals							
1000	Aluminum	µg/L	749	719	644 - 792	Acceptable	EPA 200.8
0140	Antimony †	µg/L	17.7	18.8	13.2 - 24.4	Acceptable	EPA 200.8
0001	Arsenic †	µg/L	101	102	89.3 - 114	Acceptable	EPA 200.8
0002	Barium †	µg/L	2330	2450	2080 - 2820	Acceptable	EPA 200.8
0141	Beryllium †	µg/L	6.94	6.61	5.62 - 7.60	Acceptable	EPA 200.8
0226	Boron †	µg/L	534	541	508 - 592	Acceptable	EPA 200.8
0003	Cadmium †	µg/L	13.8	12.1	9.68 - 14.5	Acceptable	EPA 200.8
0004	Chromium †	µg/L	78.8	83.8	71.2 - 96.4	Acceptable	EPA 200.8
0091	Copper †	µg/L	376	407	366 - 448	Acceptable	EPA 200.8
1070	Iron	µg/L	604	648	595 - 704	Acceptable	EPA 200.8
0005	Lead †	µg/L	78.0	66.4	46.5 - 86.3	Acceptable	EPA 200.8
0236	Manganese †	µg/L	495	471	438 - 495	Acceptable	EPA 200.8
0237	Molybdenum †	µg/L	38.8	42.7	35.6 - 49.6	Acceptable	EPA 200.8
0142	Nickel †	µg/L	146	152	129 - 175	Acceptable	EPA 200.8
0007	Selenium †	µg/L	39.2	34.1	27.3 - 40.9	Acceptable	EPA 200.8
1150	Silver	µg/L	458	488	442 - 536	Acceptable	EPA 200.8
0143	Thallium †	µg/L	12.9	8.75	6.13 - 11.4	Not Acceptable	EPA 200.8
1185	Vanadium	µg/L	466	467	435 - 497	Acceptable	EPA 200.8
0239	Zinc †	µg/L	886	959	881 - 1030	Acceptable	EPA 200.8
Mercury							
0006	Mercury †	µg/L	7.20	7.38	5.17 - 9.59	Acceptable	EPA 245.1
Inorganics							
1575	Chloride	mg/L	56.3	57.6	53.5 - 61.3	Acceptable	EPA 300.0
1610	Conductivity	µmhos/cm		510	483 - 537		
0010	Fluoride †	mg/L	5.53	5.56	5.00 - 6.12	Acceptable	EPA 300.0
0009	Nitrate as N †	mg/L	3.23	3.56	3.20 - 3.92	Acceptable	EPA 300.0
1820	Nitrate + Nitrite as N	mg/L	3.23	3.56	3.20 - 3.92	Acceptable	EPA 300.0
1125	Potassium	mg/L		21.4	19.4 - 23.5		
0145	Sulfate †	mg/L	20.9	21.4	18.7 - 23.8	Acceptable	EPA 300.0
0024	Total Dissolved Solids at 180°C †	mg/L		421	270 - 572		
Nitrite							
0092	Nitrite as N †	mg/L	1.99	1.91	1.62 - 2.20	Acceptable	EPA 300.0

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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WS-99 Final Complete Report

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Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/15/04
Study Dates: 10/08/04 - 11/22/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Pesticides							
0093	Alachlor †	µg/L	9.37	2.99	1.64 - 4.34	Not Acceptable	EPA 525.2
0256	Aldrin †	µg/L		1.94	0.826 - 2.44		
0094	Atrazine †	µg/L	15.9	5.58	3.07 - 8.09	Not Acceptable	EPA 525.2
7160	Butachlor	µg/L		0.00			
7410	Diazinon	µg/L		0.00			
0258	Dieldrin †	µg/L		0.743	0.521 - 0.933		
0011	Endrin †	µg/L		3.95	2.77 - 5.14		
0095	Heptachlor †	µg/L		2.75	1.51 - 3.99		
0096	Heptachlor epoxide (beta) †	µg/L		4.73	2.60 - 6.86		
0172	Hexachlorobenzene †	µg/L	2.17	1.08	0.489 - 1.41	Not Acceptable	EPA 525.2
0112	Hexachlorocyclopentadiene †	µg/L	4.84	4.58	0.112 - 5.84	Acceptable	EPA 525.2
0012	gamma-BHC (Lindane) †	µg/L		3.12	1.72 - 4.52		
0013	Methoxychlor †	µg/L		14.6	8.03 - 21.2		
7835	Metolachlor	µg/L		0.00			
7845	Metribuzin	µg/L		73.0	14.7 - 112		
7875	Molinate (ordram)	µg/L		67.6	33.5 - 96.0		
8035	Prometon	µg/L		66.2	41.9 - 66.2		
0259	Propachlor †	µg/L		1.91	1.10 - 2.75		
0113	Simazine †	µg/L	20.9	6.41	1.59 - 10.3	Not Acceptable	EPA 525.2
8220	Thiobencarb	µg/L		0.00			
0244	Trifluralin †	µg/L		1.56	0.598 - 1.89		
Regulated Semivolatiles #1							
5500	Acenaphthene	µg/L	< 0.25	0.00		Acceptable	EPA 550.1 UV
5505	Acenaphthylene	µg/L	< 0.13	0.00		Acceptable	EPA 550.1 UV
5555	Anthracene	µg/L	< 0.012	0.00		Acceptable	EPA 550.1 UV
5575	Benzo(a)anthracene	µg/L	1.02	1.94	0.871 - 2.50	Acceptable	EPA 550.1 UV
5585	Benzo(b)fluoranthene	µg/L	< 0.012	0.00		Acceptable	EPA 550.1 UV
5600	Benzo(k)fluoranthene	µg/L	< 0.012	0.00		Acceptable	EPA 550.1 UV
5590	Benzo(g,h,i)perylene	µg/L	0.726	1.92	0.434 - 3.01	Acceptable	EPA 550.1 UV
0122	Benzo(a)pyrene †	µg/L	0.839	1.05	0.260 - 1.37	Acceptable	EPA 550.1 UV
5670	Butylbenzylphthalate	µg/L		0.00			
5855	Chrysene	µg/L	< 0.01	0.00		Acceptable	EPA 550.1 UV
5895	Dibenz(a,h)anthracene	µg/L	< 0.05	0.00		Acceptable	EPA 550.1 UV
5925	Di-n-butylphthalate	µg/L		48.1	20.4 - 73.1		
6070	Diethylphthalate	µg/L		0.00			
6135	Dimethylphthalate	µg/L		0.00			

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-99 Final Complete Report

Tenkasi Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/15/04
Study Dates: 10/08/04 - 11/22/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Semivolatiles #1 (Cont.)							
6200	Di-n-octylphthalate	µg/L		43.8	22.4 - 57.4		
0134	bis(2-Ethylhexyl)adipate †	µg/L		47.6	20.2 - 68.4		
0136	bis(2-Ethylhexyl)phthalate †	µg/L		32.9	14.0 - 51.2		
6265	Fluoranthene	µg/L	0.338	0.423	0.118 - 0.613	Acceptable	EPA 550.1 UV
6270	Fluorene	µg/L	< 0.03	0.00		Acceptable	EPA 550.1 UV
6315	Indeno(1,2,3-cd)pyrene	µg/L	< 0.01	0.00		Acceptable	EPA 550.1 UV
5005	Naphthalene	µg/L	1.48	2.82	1.08 - 3.69	Acceptable	EPA 550.1 UV
6615	Phenanthrene	µg/L	< 0.01	0.00		Acceptable	EPA 550.1 UV
6665	Pyrene	µg/L	< 0.03	0.00		Acceptable	EPA 550.1 UV

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES® WS-102 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 EPA ID: KS00902

Report Issued: 03/24/05
Study Dates: 01/17/05 - 03/03/05

WS Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Values are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, and the National Environmental Laboratory Accreditation Conference (NELAC) program criteria as applicable. A parameter not added to the standard is given an Assigned Value of "0" per the guidelines contained in the USEPA's Criteria Document and NELAC standards.

The Acceptance Limits are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, the NELAC PT program criteria or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside of the Acceptance Limits.
No Evaluation	=	Reported Value cannot be evaluated.
Not Reported	=	Result not reported for an analyte.

The Method Description is the method the laboratory reported to ERA.

Any Performance Evaluation left blank indicates results were evaluated as 'Not Reported'.

WS Study Discussion:

ERA's WatR™ Supply Proficiency Testing Study, WS-102, has been reviewed by ERA Senior Management and certified compliant with the requirements of the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document (December 1998), the NELAC Standards Chapter 2 and Associated Appendices, applicable state PT programs, and those contained in the National Institute of Standards and Technology NVLAP Handbooks 150 and 150-19. ERA is a NIST NVLAP accredited PT Provider (Lab Code 200386-0).

This report contains data that are not covered by the NVLAP accreditation.

ERA's WatR™ Supply Study, WS-102, standards were examined for any anomalies. A full review of all accuracy verification, homogeneity and stability (VHS) data was completed. All analytical verification data for all analytes in the WS-102 standards met the acceptance criteria contained in the USEPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, and the National Voluntary Laboratory Accreditation Program Handbook 150-19 for Chemical Calibration for Providers of Proficiency Testing.

The data submitted by participating laboratories was also examined for study anomalies. There was one study anomaly found during the review of the study data. This anomaly is addressed on the following page.

WatR™ Supply Study, WS-102, reports shall not be reproduced except in their entirety and not without the permission of the participating laboratories. The report must not be used by the participating laboratories to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding ERA's WatR™ Supply Proficiency Testing Study, WS-102, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

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- Study Discussion: WS-102 Unregulated Volatiles - Bromomethane

During the review of the WS-102 Study data ERA observed a failure rate of 48.4% for bromomethane in the Unregulated Volatiles sample. As we believe that this failure rate is high, we carefully reviewed all data related to proving the efficacy of the standard including manufacturing and internal analytical verification data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'. No method biases or multi-modal distributions were found in the WS-102 bromomethane data set.

Per the USEPA Criteria Document, the acceptance limits for this compound are ±20% of the assigned value when the concentration is greater than or equal to 10.0 µg/L, as was the case with WS-102, and ±40% of the assigned value when the concentration is less than 10.0 µg/L. Of the last fifty-six PT studies conducted by ERA, when bromomethane has been present at a concentration greater than or equal to 10 µg/L, ERA has witnessed an average failure rate of 39.7%. The average failure rate when bromomethane has been present at concentrations less than 10 µg/L is 23.1%. To the best of our knowledge the USEPA never conducted a study containing bromomethane so a failure rate is not available to ERA for comparison. The USEPA criteria require that this analyte be manufactured at concentrations both above and below 10.0 µg/L, and randomly assigned in each study.

The high failure rates cause us to question the appropriateness of the acceptance limit criteria provided in the USEPA Criteria Document. On November 10, 2004, NELAC published new Field of Testing (FOT) tables for Potable and Non-Potable Water. In the FOTs for Potable Water, NELAC has addressed the issue of the inappropriate limits for the volatile gases by setting the acceptance criteria to ± 40% for all of the volatile gases. The FOTs are scheduled to be implemented in June 2005 by the PT providers.

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Study: **WS-102**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method A**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-102 Final Complete Report

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Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 03/24/05
Study Dates: 01/17/05 - 03/03/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Volatiles							
0039	Benzene †	µg/L	7.34	7.66	4.60 - 10.7	Acceptable	EPA 524.2
0037	Carbon tetrachloride †	µg/L	12.8	12.8	10.2 - 15.4	Acceptable	EPA 524.2
0049	Chlorobenzene †	µg/L	30.9	32.9	26.3 - 39.5	Acceptable	EPA 524.2
0054	1,2-Dichlorobenzene †	µg/L	8.38	8.97	5.38 - 12.6	Acceptable	EPA 524.2
0041	1,4-Dichlorobenzene †	µg/L	4.37	4.44	2.66 - 6.22	Acceptable	EPA 524.2
0035	1,2-Dichloroethane †	µg/L	6.31	6.88	4.13 - 9.63	Acceptable	EPA 524.2
0034	1,1-Dichloroethylene †	µg/L	6.95	6.85	4.11 - 9.59	Acceptable	EPA 524.2
0043	cis-1,2-Dichloroethylene †	µg/L	26.1	30.3	24.2 - 36.4	Acceptable	EPA 524.2
0042	trans-1,2-Dichloroethylene †	µg/L	8.67	9.31	5.59 - 13.0	Acceptable	EPA 524.2
0044	1,2-Dichloropropane †	µg/L	17.8	18.2	14.6 - 21.8	Acceptable	EPA 524.2
0048	Ethylbenzene †	µg/L	5.73	6.04	3.62 - 8.46	Acceptable	EPA 524.2
0055	Methylene chloride †	µg/L	14.1	15.5	12.4 - 18.6	Acceptable	EPA 524.2
0053	Styrene †	µg/L	16.2	19.4	15.5 - 23.3	Acceptable	EPA 524.2
0040	Tetrachloroethylene †	µg/L	5.95	6.49	3.89 - 9.09	Acceptable	EPA 524.2
0047	Toluene †	µg/L	10.7	11.5	9.20 - 13.8	Acceptable	EPA 524.2
0076	1,2,4-Trichlorobenzene †	µg/L	3.96	3.90	2.34 - 5.46	Acceptable	EPA 524.2
0036	1,1,1-Trichloroethane †	µg/L	9.54	9.98	5.99 - 14.0	Acceptable	EPA 524.2
0061	1,1,2-Trichloroethane †	µg/L	13.5	13.9	11.1 - 16.7	Acceptable	EPA 524.2
0038	Trichloroethylene †	µg/L	2.22	2.26	1.36 - 3.16	Acceptable	EPA 524.2
0032	Vinyl chloride †	µg/L	9.66	9.25	5.55 - 13.0	Acceptable	EPA 524.2
0090	Xylenes, total †	µg/L	18.8	19.8	15.8 - 23.8	Acceptable	EPA 524.2
Unregulated Volatiles							
0067	Bromobenzene †	µg/L	16.9	16.6	13.3 - 19.9	Acceptable	EPA 524.2
0089	Bromochloromethane †	µg/L	15.7	16.8	13.4 - 20.2	Acceptable	EPA 524.2
0069	Bromomethane †	µg/L	9.48	10.0	8.00 - 12.0	Acceptable	EPA 524.2
0079	n-Butylbenzene †	µg/L	18.3	19.7	15.8 - 23.6	Acceptable	EPA 524.2
0086	sec-Butylbenzene †	µg/L	17.1	18.3	14.6 - 22.0	Acceptable	EPA 524.2
0085	tert-Butylbenzene †	µg/L	6.09	6.08	3.65 - 8.51	Acceptable	EPA 524.2
5000	tert-Butylmethyl ether (MTBE)	µg/L	20.8	20.6	15.0 - 26.8	Acceptable	EPA 524.2
0070	Chloroethane †	µg/L	< 1.2	0.00		Acceptable	EPA 524.2
0068	Chloromethane †	µg/L	< 3.0	0.00		Acceptable	EPA 524.2
0071	2-Chlorotoluene †	µg/L	11.9	11.3	9.04 - 13.6	Acceptable	EPA 524.2
0072	4-Chlorotoluene †	µg/L	< 1.2	0.00		Acceptable	EPA 524.2
0057	Dibromomethane †	µg/L	18.2	19.8	15.8 - 23.8	Acceptable	EPA 524.2
0066	1,3-Dichlorobenzene †	µg/L	20.8	21.3	17.0 - 25.6	Acceptable	EPA 524.2
0088	Dichlorodifluoromethane †	µg/L	< 1.2	0.00		Acceptable	EPA 524.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-102 Final Complete Report

Tenkasi Viswanathan
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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 03/24/05
Study Dates: 01/17/05 - 03/03/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Unregulated Volatiles (Cont.)							
0056	1,1-Dichloroethane †	µg/L	12.4	12.5	10.0 - 15.0	Acceptable	EPA 524.2
0059	1,3-Dichloropropane †	µg/L	4.05	4.21	2.53 - 5.89	Acceptable	EPA 524.2
0060	2,2-Dichloropropane †	µg/L	6.05	6.59	3.95 - 9.23	Acceptable	EPA 524.2
0058	1,1-Dichloropropene †	µg/L	< 1.2	0.00		Acceptable	EPA 524.2
0152	cis-1,3-Dichloropropene †	µg/L	6.88	6.46	3.88 - 9.04	Acceptable	EPA 524.2
0153	trans-1,3-Dichloropropene †	µg/L	< 0.6	1.86	1.12 - 2.60	Not Acceptable	EPA 524.2
0087	Fluorotrichloromethane †	µg/L	5.53	4.86	2.92 - 6.80	Acceptable	EPA 524.2
0081	Hexachlorobutadiene †	µg/L	7.47	7.57	4.54 - 10.6	Acceptable	EPA 524.2
0084	Isopropylbenzene †	µg/L	< 1.2	0.00		Acceptable	EPA 524.2
0083	4-Isopropyltoluene †	µg/L	7.13	7.35	4.41 - 10.3	Acceptable	EPA 524.2
0078	n-Propylbenzene †	µg/L	18.5	19.0	15.2 - 22.8	Acceptable	EPA 524.2
0063	1,1,1,2-Tetrachloroethane †	µg/L	14.6	13.2	10.6 - 15.8	Acceptable	EPA 524.2
0065	1,1,2,2-Tetrachloroethane †	µg/L	9.89	9.14	5.48 - 12.8	Acceptable	EPA 524.2
0077	1,2,3-Trichlorobenzene †	µg/L	< 1.2	0.00		Acceptable	EPA 524.2
0064	1,2,3-Trichloropropane †	µg/L	< 1.2	0.00		Acceptable	EPA 524.2
0075	1,2,4-Trimethylbenzene †	µg/L	11.9	13.3	10.6 - 16.0	Acceptable	EPA 524.2
0082	1,3,5-Trimethylbenzene †	µg/L	8.9	9.42	5.65 - 13.2	Acceptable	EPA 524.2
Regulated Pesticides							
0093	Alachlor †	µg/L		17.7	9.74 - 25.7		
0256	Aldrin †	µg/L	0.518	0.492	0.196 - 0.620	Acceptable	EPA 508.1
0094	Atrazine †	µg/L		27.8	15.3 - 40.3		
7160	Butachlor	µg/L		0.00			
7410	Diazinon	µg/L		0.00			
0258	Dieldrin †	µg/L	1.71	1.80	1.26 - 2.25	Acceptable	EPA 508.1
0011	Endrin †	µg/L	4.11	3.87	2.71 - 5.03	Acceptable	EPA 508.1
0095	Heptachlor †	µg/L	0.533	0.628	0.345 - 0.911	Acceptable	EPA 508.1
0096	Heptachlor epoxide (beta) †	µg/L	2.90	3.00	1.65 - 4.35	Acceptable	EPA 508.1
0172	Hexachlorobenzene †	µg/L		2.09	0.957 - 2.67		
0112	Hexachlorocyclopentadiene †	µg/L		2.12	0.127 - 2.71		
0012	gamma-BHC (Lindane) †	µg/L	0.336	0.419	0.230 - 0.608	Acceptable	EPA 508.1
0013	Methoxychlor †	µg/L	50.8	66.0	36.3 - 95.7	Acceptable	EPA 508.1
7835	Metolachlor	µg/L		0.00			
7845	Metribuzin	µg/L		66.8	13.4 - 102		
7875	Molinate (ordram)	µg/L		27.0	13.4 - 38.3		
8035	Prometon	µg/L		0.00			
0259	Propachlor †	µg/L		1.86	1.06 - 2.69		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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WS-102 Final Complete Report

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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 03/24/05
Study Dates: 01/17/05 - 03/03/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Pesticides (Cont.)							
0113	Simazine †	µg/L		33.7	7.44 - 47.1		
8220	Thiobencarb	µg/L		0.00			
0244	Trifluralin †	µg/L		2.02	0.826 - 2.54		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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WS-103 Definitions & Study Discussion

ENVIRONMENTAL RESOURCE ASSOCIATES®

ERA Laboratory Code: A5230-01 EPA ID: KS00902

Report Issued: 04/19/05
Study Dates: 02/14/05 - 03/31/05

WS Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Values are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, and the National Environmental Laboratory Accreditation Conference (NELAC) program criteria as applicable. A parameter not added to the standard is given an Assigned Value of "0" per the guidelines contained in the USEPA's Criteria Document and NELAC standards.

The Acceptance Limits are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, the NELAC PT program criteria or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside of the Acceptance Limits.
No Evaluation	=	Reported Value cannot be evaluated.
Not Reported	=	Result not reported for an analyte.

The Method Description is the method the laboratory reported to ERA.

Any Performance Evaluation left blank indicates results were evaluated as 'Not Reported'.

WS Study Discussion:

ERA's WatR™ Supply Proficiency Testing Study, WS-103, has been reviewed by ERA Senior Management and certified compliant with the requirements of the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document (December 1998), the NELAC Standards Chapter 2 and Associated Appendices, applicable state PT programs, and those contained in the National Institute of Standards and Technology NVLAP Handbooks 150 and 150-19. ERA is a NIST NVLAP accredited PT Provider (Lab Code 200386-0).

This report contains data that are not covered by the NVLAP accreditation.

ERA's WatR™ Supply Study, WS-103, standards were examined for any anomalies. A full review of all accuracy verification, homogeneity and stability (VHS) data was completed. All analytical verification data for all analytes in the WS-103 standards met the acceptance criteria contained in the USEPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, and the National Voluntary Laboratory Accreditation Program Handbook 150-19 for Chemical Calibration for Providers of Proficiency Testing.

The data submitted by participating laboratories was also examined for study anomalies. There was one study anomaly found during the review of the study data. This anomaly is addressed on the following page.

WatR™ Supply Study, WS-103, reports shall not be reproduced except in their entirety and not without the permission of the participating laboratories. The report must not be used by the participating laboratories to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding ERA's WatR™ Supply Proficiency Testing Study, WS-103, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

- Study Discussion: Unregulated Volatiles - Chloroethane

During the statistical review of the WS-103 Unregulated Volatiles data set, ERA observed what we consider to be a high failure rate of 31.8% for chloroethane. We carefully reviewed all data related to proving the efficacy of the standard including manufacturing, internal analytical verification and stability data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'.

Per the USEPA Criteria Document, the acceptance limits for chloroethane are ±20% of the assigned value when the concentration is greater than or equal to 10.0 µg/L, as was the case with WS-103, and ±40% of the assigned value when the concentration is less than 10.0 µg/L. Of the last forty-eight PT studies conducted by ERA, when chloroethane has been present at a concentration greater than or equal to 10 µg/L, ERA has documented an average failure rate of 29.1%. The average failure rate when chloroethane has been present at a concentration less than 10 µg/L is 9.04%. To the best of our knowledge the USEPA never conducted a study containing chloroethane so a failure rate is not available for comparison. The USEPA's criteria require that this analyte be manufactured at concentrations both above and below 10.0 µg/L and randomly assigned in each study.

The high failure rate causes us to question the appropriateness of the acceptance limit criteria provided in the USEPA Criteria Document. On November 10, 2004, NELAC published new Field of Proficiency Testing (FoPT) tables for Potable and Non-Potable Water. In the FoPT tables for Potable Water, NELAC has addressed the issue of the inappropriate limits for volatile gases by setting the acceptance criteria to ± 40% for all concentrations. The FoPT tables are scheduled to be implemented in June 2005, by PT providers.

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

Study: **WS-103**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method A**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-103 Final Complete Report

Tenkasi Viswanathan
Quality Assurance Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 04/19/05
Study Dates: 02/14/05 - 03/31/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Volatiles							
0039	Benzene †	µg/L	3.66	3.28	1.97 - 4.59	Acceptable	EPA 524.2
0037	Carbon tetrachloride †	µg/L	6.38	7.63	4.58 - 10.7	Acceptable	EPA 524.2
0049	Chlorobenzene †	µg/L	21.6	20.0	16.0 - 24.0	Acceptable	EPA 524.2
0054	1,2-Dichlorobenzene †	µg/L	16.7	15.6	12.5 - 18.7	Acceptable	EPA 524.2
0041	1,4-Dichlorobenzene †	µg/L	20.6	18.8	15.0 - 22.6	Acceptable	EPA 524.2
0035	1,2-Dichloroethane †	µg/L	20.4	19.2	15.4 - 23.0	Acceptable	EPA 524.2
0034	1,1-Dichloroethylene †	µg/L	8.57	9.23	5.54 - 12.9	Acceptable	EPA 524.2
0043	cis-1,2-Dichloroethylene †	µg/L	17.4	15.2	12.2 - 18.2	Acceptable	EPA 524.2
0042	trans-1,2-Dichloroethylene †	µg/L	9.03	9.83	5.90 - 13.8	Acceptable	EPA 524.2
0044	1,2-Dichloropropane †	µg/L	5.96	5.35	3.21 - 7.49	Acceptable	EPA 524.2
0048	Ethylbenzene †	µg/L	14.4	13.8	11.0 - 16.6	Acceptable	EPA 524.2
0055	Methylene chloride †	µg/L	7.06	7.09	4.25 - 9.93	Acceptable	EPA 524.2
0053	Styrene †	µg/L	10.7	9.44	5.66 - 13.2	Acceptable	EPA 524.2
0040	Tetrachloroethylene †	µg/L	15.4	15.1	12.1 - 18.1	Acceptable	EPA 524.2
0047	Toluene †	µg/L	8.24	7.90	4.74 - 11.1	Acceptable	EPA 524.2
0076	1,2,4-Trichlorobenzene †	µg/L	14.0	13.5	10.8 - 16.2	Acceptable	EPA 524.2
0036	1,1,1-Trichloroethane †	µg/L	5.51	5.70	3.42 - 7.98	Acceptable	EPA 524.2
0061	1,1,2-Trichloroethane †	µg/L	6.38	5.35	3.21 - 7.49	Acceptable	EPA 524.2
0038	Trichloroethylene †	µg/L	12.9	12.6	10.1 - 15.1	Acceptable	EPA 524.2
0032	Vinyl chloride †	µg/L	12.7	14.3	8.58 - 20.0	Acceptable	EPA 524.2
0090	Xylenes, total †	µg/L	6.14	5.79	3.47 - 8.11	Acceptable	EPA 524.2
Unregulated Volatiles							
0067	Bromobenzene †	µg/L	11.9	11.2	8.96 - 13.4	Acceptable	EPA 524.2
0089	Bromochloromethane †	µg/L	14.3	14.6	11.7 - 17.5	Acceptable	EPA 524.2
0069	Bromomethane †	µg/L	< 3.0	0.00		Acceptable	EPA 524.2
0079	n-Butylbenzene †	µg/L	12.5	12.9	10.3 - 15.5	Acceptable	EPA 524.2
0086	sec-Butylbenzene †	µg/L	5.52	5.67	3.40 - 7.94	Acceptable	EPA 524.2
0085	tert-Butylbenzene †	µg/L	5.23	5.36	3.22 - 7.50	Acceptable	EPA 524.2
5000	tert-Butylmethyl ether (MTBE)	µg/L	< 1.2	0.00		Acceptable	EPA 524.2
0070	Chloroethane †	µg/L	12.0	12.4	9.92 - 14.9	Acceptable	EPA 524.2
0068	Chloromethane †	µg/L	8.47	6.40	3.84 - 8.96	Acceptable	EPA 524.2
0071	2-Chlorotoluene †	µg/L	10.8	10.3	8.24 - 12.4	Acceptable	EPA 524.2
0072	4-Chlorotoluene †	µg/L	20.4	18.2	14.6 - 21.8	Acceptable	EPA 524.2
0057	Dibromomethane †	µg/L	< 1.2	0.00		Acceptable	EPA 524.2
0066	1,3-Dichlorobenzene †	µg/L	24.8	24.6	19.7 - 29.5	Acceptable	EPA 524.2
0088	Dichlorodifluoromethane †	µg/L	< 1.2	0.00		Acceptable	EPA 524.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

Tenkasi Viswanathan
Quality Assurance Director
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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 04/19/05
Study Dates: 02/14/05 - 03/31/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Unregulated Volatiles (Cont.)							
0056	1,1-Dichloroethane †	µg/L	7.64	7.27	4.36 - 10.2	Acceptable	EPA 524.2
0059	1,3-Dichloropropane †	µg/L	18.7	18.4	14.7 - 22.1	Acceptable	EPA 524.2
0060	2,2-Dichloropropane †	µg/L	13.8	14.6	11.7 - 17.5	Acceptable	EPA 524.2
0058	1,1-Dichloropropene †	µg/L	< 1.2	0.00		Acceptable	EPA 524.2
0152	cis-1,3-Dichloropropene †	µg/L	< 0.6	0.00		Acceptable	EPA 524.2
0153	trans-1,3-Dichloropropene †	µg/L	< 0.6	0.00		Acceptable	EPA 524.2
0087	Fluorotrichloromethane †	µg/L	11.0	9.37	5.62 - 13.1	Acceptable	EPA 524.2
0081	Hexachlorobutadiene †	µg/L	12.5	12.3	9.84 - 14.8	Acceptable	EPA 524.2
0084	Isopropylbenzene †	µg/L	15.5	14.7	11.8 - 17.6	Acceptable	EPA 524.2
0083	4-Isopropyltoluene †	µg/L	< 1.2	0.00		Acceptable	EPA 524.2
0078	n-Propylbenzene †	µg/L	17.7	17.0	13.6 - 20.4	Acceptable	EPA 524.2
0063	1,1,1,2-Tetrachloroethane †	µg/L	2.57	2.30	1.38 - 3.22	Acceptable	EPA 524.2
0065	1,1,2,2-Tetrachloroethane †	µg/L	< 1.2	0.00		Acceptable	EPA 524.2
0077	1,2,3-Trichlorobenzene †	µg/L	19.2	18.7	15.0 - 22.4	Acceptable	EPA 524.2
0064	1,2,3-Trichloropropane †	µg/L	17.6	14.3	11.4 - 17.2	Not Acceptable	EPA 524.2
0075	1,2,4-Trimethylbenzene †	µg/L	< 1.2	0.00		Acceptable	EPA 524.2
0082	1,3,5-Trimethylbenzene †	µg/L	10.4	11.3	9.04 - 13.6	Acceptable	EPA 524.2
Regulated Pesticides							
0093	Alachlor †	µg/L		12.5	6.88 - 18.1		
0256	Aldrin †	µg/L	1.23	1.94	0.826 - 2.44	Acceptable	EPA 508.1
0094	Atrazine †	µg/L		27.2	15.0 - 39.4		
7160	Butachlor	µg/L		67.6	29.8 - 101		
7410	Diazinon	µg/L		0.00			
0258	Dieldrin †	µg/L	2.55	2.92	2.04 - 3.65	Acceptable	EPA 508.1
0011	Endrin †	µg/L	1.35	1.32	0.924 - 1.72	Acceptable	EPA 508.1
0095	Heptachlor †	µg/L	1.70	2.83	1.56 - 4.10	Acceptable	EPA 508.1
0096	Heptachlor epoxide (beta) †	µg/L	0.559	0.710	0.391 - 1.03	Acceptable	EPA 508.1
0172	Hexachlorobenzene †	µg/L		3.97	1.83 - 5.01		
0112	Hexachlorocyclopentadiene †	µg/L		3.70	0.118 - 4.72		
0012	gamma-BHC (Lindane) †	µg/L	0.978	1.25	0.688 - 1.81	Acceptable	EPA 508.1
0013	Methoxychlor †	µg/L	10.7	11.4	6.27 - 16.5	Acceptable	EPA 508.1
7835	Metolachlor	µg/L		0.00			
7845	Metribuzin	µg/L		0.00			
7875	Molinate (ordram)	µg/L		0.00			
8035	Prometon	µg/L		57.0	14.0 - 74.0		
0259	Propachlor †	µg/L		1.30	0.683 - 1.98		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-103 Final Complete Report

Tenkasi Viswanathan
Quality Assurance Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 04/19/05
Study Dates: 02/14/05 - 03/31/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Pesticides (Cont.)							
0113	Simazine †	µg/L		7.59	1.84 - 11.9		
8220	Thiobencarb	µg/L		0.00			
0244	Trifluralin †	µg/L		4.04	1.83 - 5.41		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Lab code: 200386-0

WS-105 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 EPA ID: KS00902

Report Issued: 06/16/05
Study Dates: 04/11/05 - 05/26/05

WS Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Values are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, and the National Environmental Laboratory Accreditation Conference (NELAC) program criteria as applicable. A parameter not added to the standard is given an Assigned Value of "0" per the guidelines contained in the USEPA's Criteria Document and NELAC standards.

The Acceptance Limits are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, the NELAC PT program criteria or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable	=	Reported Value falls within the Acceptance Limits.
Not Acceptable	=	Reported Value falls outside of the Acceptance Limits.
No Evaluation	=	Reported Value cannot be evaluated.
Not Reported	=	Result not reported for an analyte.

The Method Description is the method the laboratory reported to ERA.

Any Performance Evaluation left blank indicates results were evaluated as 'Not Reported'.

WS Study Discussion:

ERA's WatR™ Supply Proficiency Testing Study, WS-105, has been reviewed by ERA Senior Management and certified compliant with the requirements of the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document (December 1998), the NELAC Standards Chapter 2 and Associated Appendices, applicable state PT programs, and those contained in the National Institute of Standards and Technology NVLAP Handbooks 150 and 150-19. ERA is a NIST NVLAP accredited PT Provider (Lab Code 200386-0).

This report contains data that are not covered by the NVLAP accreditation.

ERA's WatR™ Supply Study, WS-105, standards were examined for any anomalies. A full review of all accuracy verification, homogeneity and stability (VHS) data was completed. All analytical verification data for all analytes in the WS-105 standards met the acceptance criteria contained in the USEPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, and the National Voluntary Laboratory Accreditation Program Handbook 150-19 for Chemical Calibration for Providers of Proficiency Testing.

The data submitted by participating laboratories was also examined for study anomalies. There was one study anomaly found during the review of the study data. This anomaly is addressed on the following page.

WatR™ Supply Study, WS-105, reports shall not be reproduced except in their entirety and not without the permission of the participating laboratories. The report must not be used by the participating laboratories to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding ERA's WatR™ Supply Proficiency Testing Study, WS-105, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

- Study Discussion: Unregulated Volatiles - Chloromethane

During the statistical review of the WS-105 Unregulated Volatiles data set, ERA observed what we consider to be a high failure rate of 40.5% for chloromethane. We carefully reviewed all data related to proving the efficacy of the standard including manufacturing, internal analytical verification and stability data for both accuracy and homogeneity. Our review of the data confirmed that the standard is 'fit for use'.

Per the USEPA Criteria Document, the acceptance limits for chloromethane are ± 20% of the assigned value when the concentration is greater than or equal to 10.0 µg/L, as was the case with WS-105, and ± 40% of the assigned value when the concentration is less than 10.0 µg/L. Of the last forty-eight PT studies conducted by ERA, when chloromethane has been present at a concentration greater than or equal to 10 µg/L, ERA has documented an average failure rate of 39.6%. The average failure rate when chloromethane has been present at a concentration less than 10 µg/L is 14.1%. To the best of our knowledge the USEPA never conducted a study containing chloromethane so a failure rate is not available for comparison. The USEPA's criteria require that this analyte be manufactured at concentrations both above and below 10.0 µg/L and randomly assigned in each study.

The high failure rate cause us to question the appropriateness of the acceptance limit criteria provided in the USEPA Criteria Document. On November 10, 2004, NELAC published new Field of Proficiency Testing (FoPT) tables for Potable and Non-Potable Water. In the FoPT tables for Potable Water, NELAC has addressed the issue of the inappropriate limits for volatile gases by setting the acceptance criteria to ± 40% for all concentrations. The new FoPT tables were implemented for all studies that opened June 1, 2005 or later.

NVLAP® Lab code: 200385-0

ENVIRONMENTAL
RESOURCE ASSOCIATES®

Study: **WS-105**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method A**

ACCREDITED
Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP
Lab Code: 200388-0

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-105 Final Complete Report

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QA Director
Analytical Management Labs
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Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/16/05
Study Dates: 04/11/05 - 05/26/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Metals							
1000	Aluminum	µg/L	1110	1080	973 - 1180	Acceptable	EPA 200.8
0140	Antimony †	µg/L	40.1	41.8	29.3 - 54.3	Acceptable	EPA 200.8
0001	Arsenic †	µg/L	56.9	60.4	52.8 - 67.5	Acceptable	EPA 200.8
0002	Barium †	µg/L	874	890	757 - 1020	Acceptable	EPA 200.8
0141	Beryllium †	µg/L	8.30	8.98	7.63 - 10.3	Acceptable	EPA 200.8
0226	Boron †	µg/L	758	840	783 - 926	Not Acceptable	EPA 200.8
0003	Cadmium †	µg/L	36.0	37.0	29.6 - 44.4	Acceptable	EPA 200.8
0004	Chromium †	µg/L	39.1	42.0	35.7 - 48.3	Acceptable	EPA 200.8
0091	Copper †	µg/L	265	274	247 - 301	Acceptable	EPA 200.8
1070	Iron	µg/L	1080	1080	993 - 1170	Acceptable	EPA 200.8
0005	Lead †	µg/L	55.1	54.5	38.2 - 70.9	Acceptable	EPA 200.8
0236	Manganese †	µg/L	318	306	284 - 322	Acceptable	EPA 200.8
0237	Molybdenum †	µg/L	27.7	28.8	23.9 - 33.7	Acceptable	EPA 200.8
0142	Nickel †	µg/L	113	115	97.8 - 132	Acceptable	EPA 200.8
0007	Selenium †	µg/L	33.4	32.2	25.8 - 38.6	Acceptable	EPA 200.8
1150	Silver	µg/L	92.6	99.3	89.5 - 109	Acceptable	EPA 200.8
0143	Thallium †	µg/L	9.03	8.79	6.15 - 11.4	Acceptable	EPA 200.8
1185	Vanadium	µg/L	426	430	400 - 457	Acceptable	EPA 200.8
0239	Zinc †	µg/L	1150	1150	1060 - 1230	Acceptable	EPA 200.8
Mercury							
0006	Mercury †	µg/L	2.71	3.24	2.27 - 4.21	Acceptable	EPA 245.1
Inorganics							
1575	Chloride	mg/L	84.2	83.6	78.2 - 88.8	Acceptable	EPA 300.0
1610	Conductivity	µmhos/cm	651	655	619 - 691	Acceptable	EPA 120.1
0010	Fluoride †	mg/L	4.64	4.96	4.46 - 5.46	Acceptable	EPA 300.0
0009	Nitrate as N †	mg/L	8.75	9.23	8.31 - 10.2	Acceptable	EPA 300.0
1820	Nitrate + Nitrite as N	mg/L	8.75	9.23	8.31 - 10.2	Acceptable	EPA 300.0
1125	Potassium	mg/L	35.9	36.0	32.8 - 39.3	Acceptable	EPA 200.7
0145	Sulfate †	mg/L	79.6	79.4	71.5 - 87.1	Acceptable	EPA 300.0
0024	Total Dissolved Solids at 180°C †	mg/L	372	414	265 - 563	Acceptable	EPA 160.1
Residual Chlorine							
0022	Free residual chlorine †	mg/L	2.3	2.09	1.75 - 2.43	Acceptable	EPA 330.4
1940	Total residual chlorine	mg/L	2.4	2.19	1.86 - 2.51	Acceptable	EPA 330.4

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP®

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-105 Final Complete Report

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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/16/05
Study Dates: 04/11/05 - 05/26/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Nitrite							
0092	Nitrite as N †	mg/L	1.78	1.70	1.45 - 1.96	Acceptable	EPA 300.0
Total Organic Carbon							
0263	Total organic carbon †	mg/L	3.53	3.55	3.18 - 4.11	Acceptable	EPA 415.1

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP®

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-105 Final Complete Report

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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/16/05
Study Dates: 04/11/05 - 05/26/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Volatiles							
0039	Benzene †	µg/L	14.4	14.6	11.7 - 17.5	Acceptable	EPA 524.2
0037	Carbon tetrachloride †	µg/L	11.2	11.9	9.52 - 14.3	Acceptable	EPA 524.2
0049	Chlorobenzene †	µg/L	27.8	26.1	20.9 - 31.3	Acceptable	EPA 524.2
0054	1,2-Dichlorobenzene †	µg/L	11.9	11.1	8.88 - 13.3	Acceptable	EPA 524.2
0041	1,4-Dichlorobenzene †	µg/L	19.2	18.8	15.0 - 22.6	Acceptable	EPA 524.2
0035	1,2-Dichloroethane †	µg/L	10.9	10.9	8.72 - 13.1	Acceptable	EPA 524.2
0034	1,1-Dichloroethylene †	µg/L	13.9	13.3	10.6 - 16.0	Acceptable	EPA 524.2
0043	cis-1,2-Dichloroethylene †	µg/L	33.9	33.2	26.6 - 39.8	Acceptable	EPA 524.2
0042	trans-1,2-Dichloroethylene †	µg/L	41.9	42.3	33.8 - 50.8	Acceptable	EPA 524.2
0044	1,2-Dichloropropane †	µg/L	4.33	4.31	2.59 - 6.03	Acceptable	EPA 524.2
0048	Ethylbenzene †	µg/L	11.6	11.6	9.28 - 13.9	Acceptable	EPA 524.2
0055	Methylene chloride †	µg/L	6.19	6.20	3.72 - 8.68	Acceptable	EPA 524.2
0053	Styrene †	µg/L	10.9	11.5	9.20 - 13.8	Acceptable	EPA 524.2
0040	Tetrachloroethylene †	µg/L	10.6	10.3	8.24 - 12.4	Acceptable	EPA 524.2
0047	Toluene †	µg/L	3.54	3.73	2.24 - 5.22	Acceptable	EPA 524.2
0076	1,2,4-Trichlorobenzene †	µg/L	4.26	3.74	2.24 - 5.24	Acceptable	EPA 524.2
0036	1,1,1-Trichloroethane †	µg/L	15.1	15.0	12.0 - 18.0	Acceptable	EPA 524.2
0061	1,1,2-Trichloroethane †	µg/L	3.62	3.62	2.17 - 5.07	Acceptable	EPA 524.2
0038	Trichloroethylene †	µg/L	7.22	7.18	4.31 - 10.1	Acceptable	EPA 524.2
0032	Vinyl chloride †	µg/L	3.52	3.14	1.88 - 4.40	Acceptable	EPA 524.2
0090	Xylenes, total †	µg/L	36.5	36.5	29.2 - 43.8	Acceptable	EPA 524.2
Halomethanes							
0019	Bromodichloromethane †	µg/L	48.8	43.2	34.6 - 51.8	Acceptable	EPA 524.2
0018	Bromoform †	µg/L	17.4	18.0	14.4 - 21.6	Acceptable	EPA 524.2
0020	Chlorodibromomethane †	µg/L	26.5	24.2	19.4 - 29.0	Acceptable	EPA 524.2
0017	Chloroform †	µg/L	16.1	14.8	11.8 - 17.8	Acceptable	EPA 524.2
0021	Total Trihalomethanes †	µg/L	109	100	80.0 - 120	Acceptable	EPA 524.2
Unregulated Volatiles							
0067	Bromobenzene †	µg/L	17.9	16.6	13.3 - 19.9	Acceptable	EPA 524.2
0089	Bromochloromethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0069	Bromomethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0079	n-Butylbenzene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0086	sec-Butylbenzene †	µg/L	10.2	10.7	8.56 - 12.8	Acceptable	EPA 524.2
0085	tert-Butylbenzene †	µg/L	11.5	11.8	9.44 - 14.2	Acceptable	EPA 524.2
5000	tert-Butylmethyl ether (MTBE)	µg/L	15.5	15.8	9.48 - 22.1	Acceptable	EPA 524.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-105 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/16/05
Study Dates: 04/11/05 - 05/26/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Unregulated Volatiles (Cont.)							
0070	Chloroethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0068	Chloromethane †	µg/L	16.0	16.3	13.0 - 19.6	Acceptable	EPA 524.2
0071	2-Chlorotoluene †	µg/L	11.6	11.7	9.36 - 14.0	Acceptable	EPA 524.2
0072	4-Chlorotoluene †	µg/L	13.8	14.3	11.4 - 17.2	Acceptable	EPA 524.2
0057	Dibromomethane †	µg/L	6.42	6.26	3.76 - 8.76	Acceptable	EPA 524.2
0066	1,3-Dichlorobenzene †	µg/L	24.0	23.0	18.4 - 27.6	Acceptable	EPA 524.2
0088	Dichlorodifluoromethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0056	1,1-Dichloroethane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0059	1,3-Dichloropropane †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0060	2,2-Dichloropropane †	µg/L	10.5	11.1	8.88 - 13.3	Acceptable	EPA 524.2
0058	1,1-Dichloropropene †	µg/L	10.9	12.2	9.76 - 14.6	Acceptable	EPA 524.2
0152	cis-1,3-Dichloropropene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0153	trans-1,3-Dichloropropene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0087	Fluorotrichloromethane †	µg/L	7.45	8.24	4.94 - 11.5	Acceptable	EPA 524.2
0081	Hexachlorobutadiene †	µg/L	< 0.5	0.00		Acceptable	EPA 524.2
0084	Isopropylbenzene †	µg/L	12.8	12.6	10.1 - 15.1	Acceptable	EPA 524.2
0083	4-Isopropyltoluene †	µg/L	15.3	15.5	12.4 - 18.6	Acceptable	EPA 524.2
5005	Naphthalene	µg/L	16.7	17.2	10.3 - 24.1	Acceptable	EPA 524.2
0078	n-Propylbenzene †	µg/L	10.5	10.7	8.56 - 12.8	Acceptable	EPA 524.2
0063	1,1,1,2-Tetrachloroethane †	µg/L	13.1	14.1	11.3 - 16.9	Acceptable	EPA 524.2
0065	1,1,2,2-Tetrachloroethane †	µg/L	21.3	18.7	15.0 - 22.4	Acceptable	EPA 524.2
0077	1,2,3-Trichlorobenzene †	µg/L	11.1	10.9	8.72 - 13.1	Acceptable	EPA 524.2
0064	1,2,3-Trichloropropane †	µg/L	19.5	17.2	13.8 - 20.6	Acceptable	EPA 524.2
0075	1,2,4-Trimethylbenzene †	µg/L	7.71	8.10	4.86 - 11.3	Acceptable	EPA 524.2
0082	1,3,5-Trimethylbenzene †	µg/L	10.3	10.8	8.64 - 13.0	Acceptable	EPA 524.2
Regulated Pesticides							
0093	Alachlor †	µg/L		3.02	1.66 - 4.38		
0256	Aldrin †	µg/L	1.58	1.94	0.826 - 2.44	Acceptable	EPA 508.1
0094	Atrazine †	µg/L		12.9	7.10 - 18.7		
7130	Bromacil	µg/L		0.00			
7160	Butachlor	µg/L		13.4	7.33 - 17.8		
7410	Diazinon	µg/L		0.00			
0258	Dieldrin †	µg/L	1.44	1.65	1.15 - 2.07	Acceptable	EPA 508.1
0011	Endrin †	µg/L	2.70	2.99	2.09 - 3.89	Acceptable	EPA 508.1
0095	Heptachlor †	µg/L	1.92	2.58	1.42 - 3.74	Acceptable	EPA 508.1
0096	Heptachlor epoxide (beta) †	µg/L	1.40	1.62	0.891 - 2.35	Acceptable	EPA 508.1

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-105 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/16/05
Study Dates: 04/11/05 - 05/26/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Pesticides (Cont.)							
0172	Hexachlorobenzene †	µg/L	27.1	2.65	1.22 - 3.37	Not Acceptable	EPA 508.1
0112	Hexachlorocyclopentadiene †	µg/L		3.10	0.121 - 3.96		
0012	gamma-BHC (Lindane) †	µg/L	1.92	2.70	1.49 - 3.92	Acceptable	EPA 508.1
0013	Methoxychlor †	µg/L		34.2	18.8 - 49.6		
7835	Metolachlor	µg/L		0.00			
7845	Metribuzin	µg/L		16.7	3.70 - 23.9		
7875	Molinate (ordram)	µg/L		38.4	21.1 - 55.7		
8035	Prometon	µg/L		0.00			
0259	Propachlor †	µg/L		2.80	1.71 - 3.89		
0113	Simazine †	µg/L		17.5	3.97 - 25.3		
8220	Thiobencarb	µg/L		0.00			
0244	Trifluralin †	µg/L		4.69	2.15 - 6.34		
Toxaphene							
0014	Toxaphene †	µg/L	10.0	11.9	6.55 - 17.3	Acceptable	EPA 508.1
Chlordane							
0097	Chlordane, technical †	µg/L	7.29	8.76	4.82 - 12.7	Acceptable	EPA 508.1
EDB/DBCP							
0045	Dibromochloropropane (DBCP) †	µg/L	0.997	0.900	0.540 - 1.26	Acceptable	EPA 504.1
0046	Ethylene Dibromide (EDB) †	µg/L	0.987	0.865	0.519 - 1.21	Acceptable	EPA 504.1
5180	1,2,3-Trichloropropane	µg/L	1.84	1.92	1.15 - 2.69	Acceptable	EPA 504.1
Regulated Semivolatiles #1							
5500	Acenaphthene	µg/L	0.64	1.43	0.715 - 2.15	Not Acceptable	EPA 525.2
5505	Acenaphthylene	µg/L	< 0.50	0.00		Acceptable	EPA 525.2
5555	Anthracene	µg/L	2.19	3.74	1.87 - 5.61	Acceptable	EPA 525.2
5575	Benzo(a)anthracene	µg/L	1.53	2.39	1.20 - 3.59	Acceptable	EPA 525.2
5585	Benzo(b)fluoranthene	µg/L	< 0.50	0.00		Acceptable	EPA 525.2
5600	Benzo(k)fluoranthene	µg/L	2.21	4.77	2.39 - 7.16	Not Acceptable	EPA 525.2
5590	Benzo(g,h,i)perylene	µg/L	1.56	3.82	1.91 - 5.73	Not Acceptable	EPA 525.2
0122	Benzo(a)pyrene †	µg/L	0.55	0.696	0.182 - 0.924	Acceptable	EPA 525.2
5670	Butylbenzylphthalate	µg/L	27.2	28.3	11.3 - 45.3	Acceptable	EPA 525.2
5855	Chrysene	µg/L	2.08	3.33	1.67 - 5.00	Acceptable	EPA 525.2
5895	Dibenz(a,h)anthracene	µg/L	< 0.50	0.00		Acceptable	EPA 525.2
5925	Di-n-butylphthalate	µg/L	28.8	25.6	10.2 - 41.0	Acceptable	EPA 525.2
6070	Diethylphthalate	µg/L	44.9	46.4	18.6 - 74.2	Acceptable	EPA 525.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-105 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/16/05
Study Dates: 04/11/05 - 05/26/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Semivolatiles #1 (Cont.)							
6135	Dimethylphthalate	µg/L	< 4.0	0.00		Acceptable	EPA 525.2
6200	Di-n-octylphthalate	µg/L	31.8	34.4	13.8 - 55.0	Acceptable	EPA 525.2
0134	bis(2-Ethylhexyl)adipate †	µg/L	31.1	37.5	15.5 - 54.1	Acceptable	EPA 525.2
0136	bis(2-Ethylhexyl)phthalate †	µg/L	28.5	31.6	13.5 - 49.2	Acceptable	EPA 525.2
6265	Fluoranthene	µg/L	< 0.50	0.00		Acceptable	EPA 525.2
6270	Fluorene	µg/L	2.47	4.74	2.37 - 7.11	Acceptable	EPA 525.2
6315	Indeno(1,2,3-cd)pyrene	µg/L	< 0.50	0.00		Acceptable	EPA 525.2
5005	Naphthalene	µg/L	10.9	26.0	15.6 - 36.4	Not Acceptable	EPA 525.2
6615	Phenanthrene	µg/L	2.81	5.06	2.53 - 7.59	Acceptable	EPA 525.2
6665	Pyrene	µg/L	3.14	4.58	2.29 - 6.87	Acceptable	EPA 525.2
Regulated Herbicides							
0262	Acifluorfen †	µg/L	< 1.5	35.6	14.4 - 49.0	Not Acceptable	EPA 515.1
8530	Bentazon	µg/L	32.7	48.1	18.0 - 68.7	Acceptable	EPA 515.1
8540	Chloramben	µg/L		43.6	21.8 - 65.4		
0015	2,4-D †	µg/L	14.4	27.0	13.5 - 40.5	Acceptable	EPA 515.1
8560	2,4-DB	µg/L	71.4	78.9	35.3 - 98.5	Acceptable	EPA 515.1
8550	Dacthal diacid (DCPA)	µg/L		0.00			
0115	Dalapon †	µg/L	< 1.0	31.6	0.00 - 47.1	Acceptable	EPA 515.1
0247	Dicamba †	µg/L	21.5	71.4	18.8 - 98.8	Acceptable	EPA 515.1
8600	3,5-Dichlorobenzoic acid	µg/L	< 5.0	0.00		Acceptable	EPA 515.1
8605	Dichlorprop	µg/L	< 4.7	0.00		Acceptable	EPA 515.1
0116	Dinoseb †	µg/L	28.8	44.0	1.81 - 66.3	Acceptable	EPA 515.1
6500	4-Nitrophenol	µg/L	< 1.0	0.00		Acceptable	EPA 515.1
0102	Pentachlorophenol †	µg/L	71.4	80.1	40.1 - 120	Acceptable	EPA 515.1
0117	Picloram †	µg/L	< 2.0	32.4	0.251 - 44.7	Not Acceptable	EPA 515.1
8655	2,4,5-T	µg/L	19.8	26.6	10.5 - 36.0	Acceptable	EPA 515.1
0016	2,4,5-TP (Silvex) †	µg/L	74.0	81.4	40.7 - 122	Acceptable	EPA 515.1

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-105 Final Complete Report

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QA Director
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15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/16/05
Study Dates: 04/11/05 - 05/26/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Perchlorate							
1895	Perchlorate	µg/L	6.45	7.49	6.18 - 8.24	Acceptable	EPA 314
GAD							
4770	Ethyl-t-butylether (ETBE)	µg/L	15.0	16.7	10.0 - 23.4	Acceptable	EPA 524.2
4370	T-amylmethylether (TAME)	µg/L	8.33	9.30	5.58 - 13.0	Acceptable	EPA 524.2
9375	Di-isopropylether (DIPE)	µg/L	23.4	24.0	14.4 - 33.6	Acceptable	EPA 524.2
5175	Trichlorofluoromethane (Freon 11)	µg/L	16.6	17.4	10.4 - 24.4	Acceptable	EPA 524.2
5185	Trichlorotrifluoroethane (Freon 113)	µg/L	25.2	27.7	16.6 - 38.8	Acceptable	EPA 524.2
5000	MTBE	µg/L	41.7	42.1	25.3 - 58.9	Acceptable	EPA 524.2
WS PCBs in Water							
8912	Aroclor 1016/1242	µg/L	< 0.2	0.00		Acceptable	EPA 508.1
8890	Aroclor 1232	µg/L	< 0.2	0.00		Acceptable	EPA 508.1
8900	Aroclor 1248	µg/L	1.56	1.62	0.906 - 1.93	Acceptable	EPA 508.1
8905	Aroclor 1254	µg/L	< 0.2	0.00		Acceptable	EPA 508.1
8910	Aroclor 1260	µg/L	< 0.2	0.00		Acceptable	EPA 508.1

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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Study: **WS-105**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method B**

ACCREDITED
Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP
Lab Code: 200388-0

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-105 Final Complete Report

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Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 06/16/05
Study Dates: 04/11/05 - 05/26/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Metals							
1000	Aluminum	µg/L	1030	1080	973 - 1180	Acceptable	EPA 200.7
0140	Antimony †	µg/L	41.5	41.8	29.3 - 54.3	Acceptable	EPA 200.7
0001	Arsenic †	µg/L	62.7	60.4	52.8 - 67.5	Acceptable	EPA 200.7
0002	Barium †	µg/L	904	890	757 - 1020	Acceptable	EPA 200.7
0141	Beryllium †	µg/L	7.76	8.98	7.63 - 10.3	Acceptable	EPA 200.7
0226	Boron †	µg/L		840	783 - 926		
0003	Cadmium †	µg/L	40.4	37.0	29.6 - 44.4	Acceptable	EPA 200.7
0004	Chromium †	µg/L	44.9	42.0	35.7 - 48.3	Acceptable	EPA 200.7
0091	Copper †	µg/L	271	274	247 - 301	Acceptable	EPA 200.7
1070	Iron	µg/L	1160	1080	993 - 1170	Acceptable	EPA 200.7
0005	Lead †	µg/L	61.6	54.5	38.2 - 70.9	Acceptable	EPA 200.7
0236	Manganese †	µg/L	329	306	284 - 322	Not Acceptable	EPA 200.7
0237	Molybdenum †	µg/L	29.8	28.8	23.9 - 33.7	Acceptable	EPA 200.7
0142	Nickel †	µg/L	125	115	97.8 - 132	Acceptable	EPA 200.7
0007	Selenium †	µg/L	32.1	32.2	25.8 - 38.6	Acceptable	EPA 200.7
1150	Silver	µg/L	108	99.3	89.5 - 109	Acceptable	EPA 200.7
0143	Thallium †	µg/L		8.79	6.15 - 11.4		
1185	Vanadium	µg/L	443	430	400 - 457	Acceptable	EPA 200.7
0239	Zinc †	µg/L	1250	1150	1060 - 1230	Not Acceptable	EPA 200.7
Mercury							
0006	Mercury †	µg/L	3.25	3.24	2.27 - 4.21	Acceptable	EPA 200.8
Inorganics							
1575	Chloride	mg/L	85.1	83.6	78.2 - 88.8	Acceptable	EPA 325.3
1610	Conductivity	µmhos/cm		655	619 - 691		
0010	Fluoride †	mg/L		4.96	4.46 - 5.46		
0009	Nitrate as N †	mg/L	8.84	9.23	8.31 - 10.2	Acceptable	EPA 353.2
1820	Nitrate + Nitrite as N	mg/L	8.84	9.23	8.31 - 10.2	Acceptable	EPA 353.2
1125	Potassium	mg/L		36.0	32.8 - 39.3		
0145	Sulfate †	mg/L	83.4	79.4	71.5 - 87.1	Acceptable	EPA 375.4
0024	Total Dissolved Solids at 180°C †	mg/L		414	265 - 563		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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WS-111 Definitions & Study Discussion

ENVIRONMENTAL RESOURCE ASSOCIATES®

ERA Laboratory Code: A5230-01 EPA ID: KS00902

Report Issued: 12/16/05
Study Dates: 10/14/05 - 11/28/05

WS Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Values are established per the USEPA/NELAC FoPT Tables, June 2005. A parameter not added to the standard is given an Assigned Value of "0" per the guidelines contained in the USEPA's Criteria Document and NELAC standards.

The Acceptance Limits are established per the criteria contained in the USEPA/NELAC FoPT Tables, June 2005.

The Performance Evaluation:

Acceptable	= Reported Value falls within the Acceptance Limits.
Not Acceptable	= Reported Value falls outside of the Acceptance Limits.
No Evaluation	= Reported Value cannot be evaluated.

The Method Description is the method the laboratory reported to ERA.

Any Performance Evaluation left blank indicates results were evaluated as 'Not Reported'.

WS Study Discussion:

ERA's WatR™ Supply Proficiency Testing Study, WS-111, has been reviewed by ERA Senior Management and certified compliant with the requirements of the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document (December 1998), and the criteria contained in the NELAC FoPT Tables, June 2005. ERA is a NIST NVLAP accredited PT Provider (Lab Code 200386-0).

This report contains data that are not covered by the NVLAP accreditation.

ERA's WatR™ Supply Study, WS-111, standards were examined for any anomalies. A full review of all homogeneity, stability and accuracy verification data was completed. All analytical verification data for all analytes in the WS-111 standards met the acceptance criteria contained in the USEPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, and the criteria contained in the NELAC FoPT Tables, June 2005.

The data submitted by participating laboratories was also examined for study anomalies. There were no study anomalies observed during the statistical review of the WS-111 data.

WatR™ Supply Study, WS-111, reports shall not be reproduced except in their entirety and not without the permission of the participating laboratories. The report must not be used by the participating laboratories to claim product endorsement by NVLAP or any agency of the U. S. government.

If you have any questions regarding ERA's WatR™ Supply Proficiency Testing Study, WS-111, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

Study: **WS-111**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method A**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-111 Final Complete Report

Tenkasi S. Viswanathan
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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/16/05
Study Dates: 10/14/05 - 11/28/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Metals							
1000	Aluminum	µg/L	296	290	241 - 342	Acceptable	EPA 200.8
0140	Antimony †	µg/L	20.3	21.1	14.8 - 27.4	Acceptable	EPA 200.8
0001	Arsenic †	µg/L	31.9	33.2	23.2 - 43.2	Acceptable	EPA 200.8
0002	Barium †	µg/L	892	950	808 - 1090	Acceptable	EPA 200.8
0141	Beryllium †	µg/L	2.31	2.43	2.07 - 2.79	Acceptable	EPA 200.8
0226	Boron †	µg/L	1245	1360	1190 - 1510	Acceptable	EPA 200.8
0003	Cadmium †	µg/L	19.9	21.1	16.9 - 25.3	Acceptable	EPA 200.8
0004	Chromium †	µg/L	94.6	96.1	81.7 - 111	Acceptable	EPA 200.8
0091	Copper †	µg/L	551	546	491 - 601	Acceptable	EPA 200.8
1070	Iron	µg/L	464	470	409 - 523	Acceptable	EPA 200.8
0005	Lead †	µg/L	88.8	91.0	63.7 - 118	Acceptable	EPA 200.8
0236	Manganese †	µg/L	804	782	704 - 860	Acceptable	EPA 200.8
0237	Molybdenum †	µg/L	67.9	74.9	64.8 - 83.2	Acceptable	EPA 200.8
0142	Nickel †	µg/L	288	288	245 - 331	Acceptable	EPA 200.8
0007	Selenium †	µg/L	78.4	79.1	63.3 - 94.9	Acceptable	EPA 200.8
1150	Silver	µg/L	101	103	90.1 - 115	Acceptable	EPA 200.8
0143	Thallium †	µg/L	9.20	9.21	6.45 - 12.0	Acceptable	EPA 200.8
1185	Vanadium	µg/L	627	633	570 - 696	Acceptable	EPA 200.8
0239	Zinc †	µg/L	2170	2180	1960 - 2400	Acceptable	EPA 200.8
Mercury							
0006	Mercury †	µg/L	2.55	2.65	1.86 - 3.45	Acceptable	EPA 245.1
Inorganics							
1575	Chloride	mg/L	82.0	82.9	74.6 - 91.2	Acceptable	EPA 300.0
1610	Conductivity	µmhos/cm	506	495	446 - 545	Acceptable	EPA 120.1
0010	Fluoride †	mg/L	3.44	3.52	3.17 - 3.87	Acceptable	EPA 300.0
0009	Nitrate as N †	mg/L	3.38	3.93	3.54 - 4.32	Not Acceptable	EPA 300.0
1820	Nitrate + Nitrite as N	mg/L	3.38	3.93	3.48 - 4.32	Not Acceptable	EPA 300.0
1125	Potassium	mg/L	18.5	18.2	15.7 - 21.2	Acceptable	EPA 200.7
0145	Sulfate †	mg/L	17.7	18.3	15.0 - 21.1	Acceptable	EPA 300.0
0024	Total Dissolved Solids at 180°C †	mg/L	341	349	226 - 472	Acceptable	EPA 160.1
Alkalinity & Sodium							
0027	Alkalinity (as CaCO3) †	mg/L	40.1	39.3	35.4 - 43.4	Acceptable	EPA 310.1
0029	Sodium †	mg/L	17.2	16.9	14.9 - 18.6	Acceptable	EPA 200.7

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NVLAP®

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-111 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/16/05
Study Dates: 10/14/05 - 11/28/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Residual Chlorine							
0022	Free residual chlorine †	mg/L	0.5	0.612	0.468 - 0.757	Acceptable	EPA 330.4
1940	Total residual chlorine	mg/L	0.6	0.612	0.506 - 0.709	Acceptable	EPA 330.4
Nitrite							
0092	Nitrite as N †	mg/L	0.912	0.869	0.739 - 0.999	Acceptable	EPA 300.0
Nutrients							
0261	ortho-Phosphate as P †	mg/L	4.80	4.20	3.71 - 4.72	Not Acceptable	EPA 300.0
Cyanide							
0146	Cyanide †	mg/L	0.314	0.389	0.292 - 0.486	Acceptable	EPA 335.2
Total Organic Carbon							
0263	Total organic carbon †	mg/L	2.07	2.01	1.63 - 2.45	Acceptable	EPA 415.1
Hardness							
1755	Total Hardness as CaCO3	mg/L	164	150	134 - 168	Acceptable	SM 2340 B
0025	Calcium Hardness as CaCO3 †	mg/L	88.7	81.4	72.6 - 91.8	Acceptable	SM 2340 B
1035	Calcium	mg/L	35.5	32.6	29.1 - 36.8	Acceptable	EPA 200.7
1085	Magnesium	mg/L	18.4	16.6	14.9 - 18.5	Acceptable	EPA 200.7

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Volatiles							
0039	Benzene †	µg/L	12.1	12.8	10.2 - 15.4	Acceptable	EPA 524.2
0037	Carbon tetrachloride †	µg/L	10.2	10.6	8.48 - 12.7	Acceptable	EPA 524.2
0049	Chlorobenzene †	µg/L	16.9	16.1	12.9 - 19.3	Acceptable	EPA 524.2
0054	1,2-Dichlorobenzene †	µg/L	14.0	14.2	11.4 - 17.0	Acceptable	EPA 524.2
0041	1,4-Dichlorobenzene †	µg/L	14.4	13.9	11.1 - 16.7	Acceptable	EPA 524.2
0035	1,2-Dichloroethane †	µg/L	6.72	6.69	4.01 - 9.37	Acceptable	EPA 524.2
0034	1,1-Dichloroethylene †	µg/L	8.33	7.03	4.22 - 9.84	Acceptable	EPA 524.2
0043	cis-1,2-Dichloroethylene †	µg/L	19.4	18.9	15.1 - 22.7	Acceptable	EPA 524.2
0042	trans-1,2-Dichloroethylene †	µg/L	11.9	11.5	9.20 - 13.8	Acceptable	EPA 524.2
0044	1,2-Dichloropropane †	µg/L	12.4	12.8	10.2 - 15.4	Acceptable	EPA 524.2
0048	Ethylbenzene †	µg/L	19.0	19.4	15.5 - 23.3	Acceptable	EPA 524.2
0055	Methylene chloride (Dichloromethane) †	µg/L	18.9	18.4	14.7 - 22.1	Acceptable	EPA 524.2
0053	Styrene †	µg/L	5.81	6.05	3.63 - 8.47	Acceptable	EPA 524.2
0040	Tetrachloroethylene †	µg/L	13.6	14.4	11.5 - 17.3	Acceptable	EPA 524.2
0047	Toluene †	µg/L	18.4	19.8	15.8 - 23.8	Acceptable	EPA 524.2
0076	1,2,4-Trichlorobenzene †	µg/L	5.16	6.32	3.79 - 8.85	Acceptable	EPA 524.2
0036	1,1,1-Trichloroethane †	µg/L	11.3	11.7	9.36 - 14.0	Acceptable	EPA 524.2
0061	1,1,2-Trichloroethane †	µg/L	3.71	3.98	2.39 - 5.57	Acceptable	EPA 524.2
0038	Trichloroethylene †	µg/L	9.64	10.0	8.00 - 12.0	Acceptable	EPA 524.2
0032	Vinyl chloride †	µg/L	25.4	27.1	16.3 - 37.9	Acceptable	EPA 524.2
0090	Xylenes, total †	µg/L	14.9	16.2	13.0 - 19.4	Acceptable	EPA 524.2
Halomethanes							
0019	Bromodichloromethane †	µg/L	20.5	20.1	16.5 - 24.5	Acceptable	EPA 524.2
0018	Bromoform †	µg/L	40.6	42.3	32.0 - 51.8	Acceptable	EPA 524.2
0020	Chlorodibromomethane †	µg/L	34.6	38.1	30.3 - 45.5	Acceptable	EPA 524.2
0017	Chloroform †	µg/L	11.4	11.8	9.64 - 14.6	Acceptable	EPA 524.2
0021	Total Trihalomethanes †	µg/L	107	112	89.6 - 134	Acceptable	EPA 524.2
Unregulated Volatiles							
0067	Bromobenzene †	µg/L	29.9	31.6	25.3 - 37.9	Acceptable	EPA 524.2
0089	Bromochloromethane †	µg/L	24.2	25.9	20.7 - 31.1	Acceptable	EPA 524.2
0069	Bromomethane †	µg/L	11.9	11.2	6.72 - 15.7	Acceptable	EPA 524.2
0079	n-Butylbenzene †	µg/L	16.8	18.3	14.6 - 22.0	Acceptable	EPA 524.2
0086	sec-Butylbenzene †	µg/L	32.6	33.5	26.8 - 40.2	Acceptable	EPA 524.2
0085	tert-Butylbenzene †	µg/L	< 2.0	0.00		Acceptable	EPA 524.2

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WS-111 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/16/05
Study Dates: 10/14/05 - 11/28/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Unregulated Volatiles (Cont.)							
5000	tert-Butylmethyl ether (MTBE)	µg/L	36.0	34.0	20.4 - 47.6	Acceptable	EPA 524.2
0070	Chloroethane †	µg/L	14.9	16.7	10.0 - 23.4	Acceptable	EPA 524.2
0068	Chloromethane †	µg/L	16.7	16.7	10.0 - 23.4	Acceptable	EPA 524.2
0071	2-Chlorotoluene †	µg/L	36.1	41.2	33.0 - 49.4	Acceptable	EPA 524.2
0072	4-Chlorotoluene †	µg/L	36.2	43.1	34.5 - 51.7	Acceptable	EPA 524.2
0057	Dibromomethane †	µg/L	< 2.0	0.00		Acceptable	EPA 524.2
0066	1,3-Dichlorobenzene †	µg/L	< 2.0	0.00		Acceptable	EPA 524.2
0088	Dichlorodifluoromethane †	µg/L	< 2.0	0.00		Acceptable	EPA 524.2
0056	1,1-Dichloroethane †	µg/L	27.8	31.2	25.0 - 37.4	Acceptable	EPA 524.2
0059	1,3-Dichloropropane †	µg/L	< 2.0	0.00		Acceptable	EPA 524.2
0060	2,2-Dichloropropane †	µg/L	< 2.0	0.00		Acceptable	EPA 524.2
0058	1,1-Dichloropropene †	µg/L	< 2.0	0.00		Acceptable	EPA 524.2
0152	cis-1,3-Dichloropropene †	µg/L	40.8	45.7	36.6 - 54.8	Acceptable	EPA 524.2
0153	trans-1,3-Dichloropropene †	µg/L	13.3	14.8	8.88 - 20.7	Acceptable	EPA 524.2
0087	Fluorotrichloromethane †	µg/L	14.9	0.00		Not Acceptable	EPA 524.2
0081	Hexachlorobutadiene †	µg/L	24.1	25.2	20.2 - 30.2	Acceptable	EPA 524.2
0084	Isopropylbenzene †	µg/L	49.2	48.1	38.5 - 57.7	Acceptable	EPA 524.2
0083	4-Isopropyltoluene †	µg/L	17.8	16.2	13.0 - 19.4	Acceptable	EPA 524.2
5005	Naphthalene	µg/L	13.2	12.2	7.32 - 17.1	Acceptable	EPA 524.2
0078	n-Propylbenzene †	µg/L	10.2	9.67	5.80 - 13.5	Acceptable	EPA 524.2
0063	1,1,1,2-Tetrachloroethane †	µg/L	46.3	49.0	39.2 - 58.8	Acceptable	EPA 524.2
0065	1,1,2,2-Tetrachloroethane †	µg/L	30.4	34.5	27.6 - 41.4	Acceptable	EPA 524.2
0077	1,2,3-Trichlorobenzene †	µg/L	4.67	5.61	3.37 - 7.85	Acceptable	EPA 524.2
0064	1,2,3-Trichloropropane †	µg/L	< 2.0	0.00		Acceptable	EPA 524.2
0075	1,2,4-Trimethylbenzene †	µg/L	13.9	14.3	8.58 - 20.0	Acceptable	EPA 524.2
0082	1,3,5-Trimethylbenzene †	µg/L	9.94	9.54	5.72 - 13.4	Acceptable	EPA 524.2
Regulated Pesticides							
0093	Alachlor †	µg/L	6.05	7.18	3.95 - 10.4	Acceptable	EPA 508.1
0256	Aldrin †	µg/L	0.773	1.04	0.435 - 1.31	Acceptable	EPA 508.1
0094	Atrazine †	µg/L		7.22	3.97 - 10.5		
7130	Bromacil	µg/L		0.00			
7160	Butachlor	µg/L		54.1	28.4 - 68.3		
7410	Diazinon	µg/L		0.00			
0258	Dieldrin †	µg/L	1.94	1.91	1.19 - 2.50	Acceptable	EPA 508.1

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ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-111 Final Complete Report

Tenkasi S. Viswanathan
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15130 S Keeler
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EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/16/05
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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Pesticides (Cont.)							
0011	Endrin †	µg/L	4.08	4.57	3.20 - 5.94	Acceptable	EPA 508.1
0095	Heptachlor †	µg/L	1.87	2.14	1.18 - 3.10	Acceptable	EPA 508.1
0096	Heptachlor epoxide (beta) †	µg/L	2.61	3.23	1.78 - 4.68	Acceptable	EPA 508.1
0172	Hexachlorobenzene †	µg/L	0.652	0.843	0.379 - 1.12	Acceptable	EPA 508.1
0112	Hexachlorocyclopentadiene †	µg/L		28.2	5.38 - 39.6		
0012	gamma-BHC (Lindane) †	µg/L	1.03	2.20	1.21 - 3.19	Not Acceptable	EPA 508.1
0013	Methoxychlor †	µg/L	52.8	58.6	32.2 - 85.0	Acceptable	EPA 508.1
7835	Metolachlor	µg/L	37.5	56.1	28.6 - 69.7	Acceptable	EPA 508.1
7845	Metribuzin	µg/L		29.0	6.25 - 40.8		
7875	Molinate (ordram)	µg/L		17.1	9.41 - 24.8		
8035	Prometon	µg/L		0.00			
0259	Propachlor †	µg/L	3.32	3.86	2.31 - 5.31	Acceptable	EPA 508.1
0113	Simazine †	µg/L	5.43	17.0	3.86 - 24.6	Acceptable	EPA 508.1
8220	Thiobencarb	µg/L		0.00			
0244	Trifluralin †	µg/L	1.65	2.91	1.47 - 3.71	Acceptable	EPA 508.1
Toxaphene							
0014	Toxaphene †	µg/L	12.6	15.2	8.36 - 22.0	Acceptable	EPA 508.1
Chlordane							
0097	Chlordane, technical †	µg/L	6.78	8.98	4.94 - 13.0	Acceptable	EPA 508.1
EDB/DBCP							
0045	Dibromochloropropane (DBCP) †	µg/L	0.786	0.900	0.540 - 1.26	Acceptable	EPA 504.1
0046	Ethylene Dibromide (EDB) †	µg/L	1.68	1.73	1.04 - 2.42	Acceptable	EPA 504.1
5180	1,2,3-Trichloropropane	µg/L	1.37	1.65	0.990 - 2.31	Acceptable	EPA 504.1
Regulated Semivolatiles #1							
5500	Acenaphthene	µg/L	0.95	1.49	0.745 - 2.24	Acceptable	EPA 525.2
5505	Acenaphthylene	µg/L	4.45	6.34	3.17 - 9.51	Acceptable	EPA 525.2
5555	Anthracene	µg/L	< 0.50	0.00		Acceptable	EPA 525.2
5575	Benzo(a)anthracene	µg/L	4.09	5.71	2.86 - 8.57	Acceptable	EPA 525.2
5585	Benzo(b)fluoranthene	µg/L	5.53	6.69	3.35 - 10.0	Acceptable	EPA 525.2
5600	Benzo(k)fluoranthene	µg/L	< 0.50	0.00		Acceptable	EPA 525.2
5590	Benzo(g,h,i)perylene	µg/L	1.89	3.34	1.67 - 5.01	Acceptable	EPA 525.2
0122	Benzo(a)pyrene †	µg/L	0.61	0.847	0.402 - 1.08	Acceptable	EPA 525.2
5670	Butylbenzylphthalate	µg/L	29.0	43.7	17.5 - 69.9	Acceptable	EPA 525.2

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Semivolatiles #1 (Cont.)							
5855	Chrysene	µg/L	5.17	8.50	4.25 - 12.8	Acceptable	EPA 525.2
5895	Dibenz(a,h)anthracene	µg/L	3.17	6.20	3.10 - 9.30	Acceptable	EPA 525.2
5925	Di-n-butylphthalate	µg/L	34.9	43.9	17.6 - 70.2	Acceptable	EPA 525.2
6070	Diethylphthalate	µg/L	30.9	46.4	18.6 - 74.2	Acceptable	EPA 525.2
6135	Dimethylphthalate	µg/L	12.2	18.9	7.56 - 30.2	Acceptable	EPA 525.2
6200	Di-n-octylphthalate	µg/L	26.3	37.5	15.0 - 60.0	Acceptable	EPA 525.2
0134	bis(2-Ethylhexyl)adipate †	µg/L	32.0	47.6	20.2 - 68.4	Acceptable	EPA 525.2
0136	bis(2-Ethylhexyl)phthalate †	µg/L	13.6	19.0	7.74 - 29.4	Acceptable	EPA 525.2
6265	Fluoranthene	µg/L	4.82	5.81	2.91 - 8.72	Acceptable	EPA 525.2
6270	Fluorene	µg/L	1.49	1.96	0.980 - 2.94	Acceptable	EPA 525.2
6315	Indeno(1,2,3-cd)pyrene	µg/L	1.05	0.00		Not Acceptable	EPA 525.2
5005	Naphthalene	µg/L	21.3	34.7	20.8 - 48.6	Acceptable	EPA 525.2
6615	Phenanthrene	µg/L	2.46	3.17	1.59 - 4.76	Acceptable	EPA 525.2
6665	Pyrene	µg/L	4.41	6.11	3.06 - 9.17	Acceptable	EPA 525.2
Regulated Herbicides							
0262	Acifluorfen †	µg/L	7.81	40.2	17.7 - 53.9	Not Acceptable	EPA 515.1
8530	Bentazon	µg/L	8.18	41.5	15.2 - 59.6	Not Acceptable	EPA 515.1
8540	Chloramben	µg/L	9.10	62.2	31.1 - 93.3	Not Acceptable	EPA 515.1
0015	2,4-D †	µg/L	0.64	19.4	9.70 - 29.1	Not Acceptable	EPA 515.1
8560	2,4-DB	µg/L	2.56	73.5	32.8 - 92.1	Not Acceptable	EPA 515.1
8550	Dacthal diacid (DCPA)	µg/L	2.82	21.6	2.16 - 35.3	Acceptable	EPA 515.1
0115	Dalapon †	µg/L	15.1	146	14.6 - 197	Acceptable	EPA 515.1
0247	Dicamba †	µg/L	10.6	58.7	15.6 - 81.5	Not Acceptable	EPA 515.1
8600	3,5-Dichlorobenzoic acid	µg/L	5.13	19.5	9.75 - 29.3	Not Acceptable	EPA 515.1
8605	Dichlorprop	µg/L	2.84	20.3	11.0 - 24.1	Not Acceptable	EPA 515.1
0116	Dinoseb †	µg/L	6.02	35.2	7.30 - 49.7	Not Acceptable	EPA 515.1
6500	4-Nitrophenol	µg/L	9.68	42.9	4.29 - 64.4	Acceptable	EPA 515.1
0102	Pentachlorophenol †	µg/L	1.57	6.16	3.08 - 9.24	Not Acceptable	EPA 515.1
0117	Picloram †	µg/L	2.92	27.5	6.26 - 38.9	Not Acceptable	EPA 515.1
8655	2,4,5-T	µg/L	5.64	44.3	17.5 - 58.4	Not Acceptable	EPA 515.1
0016	2,4,5-TP (Silvex) †	µg/L	7.22	48.4	24.2 - 72.6	Not Acceptable	EPA 515.1
Haloacetic Acids							
0160	Bromoacetic acid †	µg/L	18.2	26.6	16.9 - 33.3	Acceptable	EPA 552.2
0250	Bromochloroacetic acid †	µg/L	33.6	43.4	26.0 - 60.8	Acceptable	EPA 552.2

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Haloacetic Acids (Cont.)							
0161	Chloroacetic acid †	µg/L	10.5	14.2	9.24 - 18.4	Acceptable	EPA 552.2
0157	Dibromoacetic acid †	µg/L	14.9	24.4	13.8 - 28.0	Acceptable	EPA 552.2
0158	Dichloroacetic acid †	µg/L	38.2	47.6	28.9 - 57.1	Acceptable	EPA 552.2
0162	Trichloroacetic acid †	µg/L	39.7	44.6	27.8 - 54.6	Acceptable	EPA 552.2

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Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Perchlorate							
1895	Perchlorate	µg/L	8.46	9.96	8.22 - 11.0	Acceptable	EPA 314
Chromium (VI)							
1045	Chromium (VI)	µg/L	33	33.2	29.8 - 36.5	Acceptable	EPA 7196A
Uranium							
3035	Uranium	µg/L	40.7	42.0	34.2 - 49.8	Acceptable	EPA 200.8
GAD							
4770	Ethyl-t-butylether (ETBE)	µg/L	25.9	31.3	18.8 - 43.8	Acceptable	EPA 524.2
4370	T-amylmethylether (TAME)	µg/L	24.4	29.0	17.4 - 40.6	Acceptable	EPA 524.2
9375	Di-isopropylether (DIPE)	µg/L	16.4	18.5	11.1 - 25.9	Acceptable	EPA 524.2
5175	Trichlorofluoromethane (Freon 11)	µg/L	49.6	41.7	25.0 - 58.4	Acceptable	EPA 524.2
5185	Trichlorotrifluoroethane (Freon 113)	µg/L	5.75	5.81	3.49 - 8.13	Acceptable	EPA 524.2
5000	MTBE	µg/L	16.0	17.7	10.6 - 24.8	Acceptable	EPA 524.2
WS PCBs in Water							
8912	Aroclor 1016	µg/L	< 0.1	0.00		Acceptable	EPA 508.1
8885	Aroclor 1221	µg/L	< 0.1	0.00		Acceptable	EPA 508.1
8890	Aroclor 1232	µg/L	< 0.1	0.00		Acceptable	EPA 508.1
8912	Aroclor 1242	µg/L	< 0.1	0.00		Acceptable	EPA 508.1
8900	Aroclor 1248	µg/L	< 0.1	0.00		Acceptable	EPA 508.1
8905	Aroclor 1254	µg/L	4.58	3.63	2.18 - 4.31	Not Acceptable	EPA 508.1
8910	Aroclor 1260	µg/L	< 0.1	0.00		Acceptable	EPA 508.1
Tert-Butyl Alcohol							
4420	Tert-Butyl Alcohol	µg/L	44.6	37.3	22.4 - 52.2	Acceptable	EPA 524.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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Study: **WS-111**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method B**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-111 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/16/05
Study Dates: 10/14/05 - 11/28/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Metals							
1000	Aluminum	µg/L	256	290	241 - 342	Acceptable	EPA 200.7
0140	Antimony †	µg/L	20.2	21.1	14.8 - 27.4	Acceptable	EPA 200.7
0001	Arsenic †	µg/L	31.6	33.2	23.2 - 43.2	Acceptable	EPA 200.7
0002	Barium †	µg/L	909	950	808 - 1090	Acceptable	EPA 200.7
0141	Beryllium †	µg/L	2.62	2.43	2.07 - 2.79	Acceptable	EPA 200.7
0226	Boron †	µg/L	1270	1360	1190 - 1510	Acceptable	EPA 200.7
0003	Cadmium †	µg/L	20.7	21.1	16.9 - 25.3	Acceptable	EPA 200.7
0004	Chromium †	µg/L	96.6	96.1	81.7 - 111	Acceptable	EPA 200.7
0091	Copper †	µg/L	545	546	491 - 601	Acceptable	EPA 200.7
1070	Iron	µg/L	470	470	409 - 523	Acceptable	EPA 200.7
0005	Lead †	µg/L	99.0	91.0	63.7 - 118	Acceptable	EPA 200.7
0236	Manganese †	µg/L	795	782	704 - 860	Acceptable	EPA 200.7
0237	Molybdenum †	µg/L	72.1	74.9	64.8 - 83.2	Acceptable	EPA 200.7
0142	Nickel †	µg/L	262	288	245 - 331	Acceptable	EPA 200.7
0007	Selenium †	µg/L	82.0	79.1	63.3 - 94.9	Acceptable	EPA 200.7
1150	Silver	µg/L	108	103	90.1 - 115	Acceptable	EPA 200.7
0143	Thallium †	µg/L	9.68	9.21	6.45 - 12.0	Acceptable	EPA 200.7
1185	Vanadium	µg/L	612	633	570 - 696	Acceptable	EPA 200.7
0239	Zinc †	µg/L	2250	2180	1960 - 2400	Acceptable	EPA 200.7
Mercury							
0006	Mercury †	µg/L	2.52	2.65	1.86 - 3.45	Acceptable	EPA 200.8
Inorganics							
1575	Chloride	mg/L	88.6	82.9	74.6 - 91.2	Acceptable	EPA 325.3
1610	Conductivity	µmhos/cm		495	446 - 545		
0010	Fluoride †	mg/L		3.52	3.17 - 3.87		
0009	Nitrate as N †	mg/L	2.82	3.93	3.54 - 4.32	Not Acceptable	EPA 353.2
1820	Nitrate + Nitrite as N	mg/L	2.82	3.93	3.48 - 4.32	Not Acceptable	EPA 353.2
1125	Potassium	mg/L		18.2	15.7 - 21.2		
0145	Sulfate †	mg/L	18.9	18.3	15.0 - 21.1	Acceptable	EPA 375.4
0024	Total Dissolved Solids at 180°C †	mg/L		349	226 - 472		
Nitrite							
0092	Nitrite as N †	mg/L	0.751	0.869	0.739 - 0.999	Acceptable	EPA 353.2

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP®

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-111 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/16/05
Study Dates: 10/14/05 - 11/28/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Nutrients							
0261	ortho-Phosphate as P †	mg/L	4.60	4.20	3.71 - 4.72	Acceptable	EPA 365.3

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP®

ENVIRONMENTAL RESOURCE ASSOCIATES®

WS-111 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/16/05
Study Dates: 10/14/05 - 11/28/05

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Regulated Pesticides							
0093	Alachlor †	µg/L	7.48	7.18	3.95 - 10.4	Acceptable	EPA 525.2
0256	Aldrin †	µg/L		1.04	0.435 - 1.31		
0094	Atrazine †	µg/L	6.71	7.22	3.97 - 10.5	Acceptable	EPA 525.2
7130	Bromacil	µg/L		0.00			
7160	Butachlor	µg/L		54.1	28.4 - 68.3		
7410	Diazinon	µg/L		0.00			
0258	Dieldrin †	µg/L		1.91	1.19 - 2.50		
0011	Endrin †	µg/L		4.57	3.20 - 5.94		
0095	Heptachlor †	µg/L		2.14	1.18 - 3.10		
0096	Heptachlor epoxide (beta) †	µg/L		3.23	1.78 - 4.68		
0172	Hexachlorobenzene †	µg/L	0.63	0.843	0.379 - 1.12	Acceptable	EPA 525.2
0112	Hexachlorocyclopentadiene †	µg/L	23.5	28.2	5.38 - 39.6	Acceptable	EPA 525.2
0012	gamma-BHC (Lindane) †	µg/L		2.20	1.21 - 3.19		
0013	Methoxychlor †	µg/L		58.6	32.2 - 85.0		
7835	Metolachlor	µg/L		56.1	28.6 - 69.7		
7845	Metribuzin	µg/L		29.0	6.25 - 40.8		
7875	Molinate (ordram)	µg/L		17.1	9.41 - 24.8		
8035	Prometon	µg/L		0.00			
0259	Propachlor †	µg/L		3.86	2.31 - 5.31		
0113	Simazine †	µg/L		17.0	3.86 - 24.6		
8220	Thiobencarb	µg/L		0.00			
0244	Trifluralin †	µg/L		2.91	1.47 - 3.71		

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

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ENVIRONMENTAL
RESOURCE ASSOCIATES®
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April 7, 2005

David Caldwell
State of Oklahoma
Department of Environmental Quality
Laboratory Certification Program
PO Box 1677
Oklahoma City, OK 73101-1677

Fax: 405-702-1001

Dear David,

On March 29, 2005, Analytical Management Labs located in Olathe, Kansas, participated in ERA's QuiK™ Response Performance Evaluation Program. The following results were reported to ERA by Analytical Management Labs for the PE standard, lot 032905A. The Certified Values and the QuiK™ Response Acceptance Limits were not available to Analytical Management Labs. This report will be released to Analytical Management Labs.

If you have any questions, please contact either myself, or Shawn Kassner, Proficiency Testing Program Manager, at 1-800-372-0122.

Sincerely,

Jennifer Luzader
QuiK™Response Coordinator

Cc: Project File Number 032905A

ENVIRONMENTAL
RESOURCE ASSOCIATES®
The Industry Standard™

April 7, 2005

Maren Farr\Jack McKenzie
State of Kansas
Department of Health and Environment
Forbes Field
Building 740
Topeka, KS 66620-0001

Fax: 785-296-8068

Dear Maren,

On March 29, 2005, Analytical Management Labs located in Olathe, Kansas, participated in ERA's QuiK™ Response Performance Evaluation Program. The following results were reported to ERA by Analytical Management Labs for the PE standard, lot 032905A. The Certified Values and the QuiK™ Response Acceptance Limits were not available to Analytical Management Labs. This report will be released to Analytical Management Labs.

If you have any questions, please contact either myself, or Shawn Kassner, Proficiency Testing Program Manager, at 1-800-372-0122.

Sincerely,

Jennifer Luzader
QuiK™Response Coordinator

Cc: Project File Number 032905A

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QuiK™ Response PE Standards

Final Report

Gasoline in Water

Customer: Analytical Management Labs
Lot Number: 032905A-1
State ID Number: KS00902
Method: OK-DEQ-GRO-8015

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
Unleaded gasoline	µg/L	4040	3880	2110 - 6190	Acceptable
Benzene in gasoline	µg/L		38.8	21.3 - 52.5	
Ethylbenzene in gasoline	µg/L		97.4	58.3 - 116	
Toluene in gasoline	µg/L		341	172 - 426	
Total Xylenes in gasoline	µg/L		543	273 - 672	

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

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QuiK™ Response PE Standards

Final Report

Gasoline in Water

Customer: Analytical Management Labs
Lot Number: 032905A-2
State ID Number: KS00902
Method: OK-DEQ-GRO-8015

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
Unleaded gasoline	µg/L	2210	2310	30.0 - 4830	Acceptable
Benzene in gasoline	µg/L		23.1	7.80 - 37.8	
Ethylbenzene in gasoline	µg/L		58.0	26.9 - 80.7	
Toluene in gasoline	µg/L		203	98.9 - 277	
Total Xylenes in gasoline	µg/L		323	137 - 455	

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

ENVIRONMENTAL RESOURCE ASSOCIATES®
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QuiK™ Response PE Standards

Final Report

Gasoline in Soil

Customer: Analytical Management Labs
Lot Number: 032905A-1
State ID Number: KS00902
Method: OK-DEQ-GRO-8015

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
Unleaded gasoline	mg\kg	1220	1550	156 - 2420	Acceptable
Benzene in gasoline	mg\kg		15.5	0.00 - 18.9	
Ethylbenzene in gasoline	mg\kg		38.9	13.1 - 49.3	
Toluene in gasoline	mg\kg		136	81.2 - 155	
Total Xylenes in gasoline	mg\kg		217	84.1 - 242	

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

ENVIRONMENTAL RESOURCE ASSOCIATES®
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QuiK™ Response PE Standards

Final Report

Gasoline in Soil

Customer: Analytical Management Labs
Lot Number: 032905A-2
State ID Number: KS00902
Method: OK-DEQ-GRO-8015

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
Unleaded gasoline	mg\kg	565	709	0.00 - 1130	Acceptable
Benzene in gasoline	mg\kg		7.09	0.00 - 7.09	
Ethylbenzene in gasoline	mg\kg		17.8	0.00 - 24.4	
Toluene in gasoline	mg\kg		62.2	0.00 - 72.0	
Total Xylenes in gasoline	mg\kg		99.3	0.00 - 133	

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

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QuiK™ Response PE Standards

Final Report

BTEX in Water

Customer: Analytical Management Labs
Lot Number: 032905A-1
State ID Number: KS00902
Method: OK-GRO-8021B

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
Benzene	µg/L	53.5	61.3	36.6 - 77.6	Acceptable
Ethylbenzene	µg/L	132	166	81.4 - 221	Acceptable
Toluene	µg/L	149	184	104 - 234	Acceptable
Total Xylenes	µg/L	200	226	119 - 295	Acceptable
t-Butylmethylether (MTBE)	µg/L	44.7	53.4	28.9 - 62.1	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

QuiK™ Response PE Standards

Final Report

BTEX in Water

Customer: Analytical Management Labs
Lot Number: 032905A-1
State ID Number: KS00902
Method: SW-8260B

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
Benzene	µg/L	58.8	61.3	36.6 - 77.6	Acceptable
Ethylbenzene	µg/L	183	166	81.4 - 221	Acceptable
Toluene	µg/L	200	184	104 - 234	Acceptable
Total Xylenes	µg/L	238	226	119 - 295	Acceptable
t-Butylmethylether (MTBE)	µg/L	43.0	53.4	28.9 - 62.1	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

QuiK™ Response PE Standards

Final Report

BTEX in Water

Customer: Analytical Management Labs
Lot Number: 032905A-2
State ID Number: KS00902
Method: OK-GRO-8021B

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
Benzene	µg/L	144	163	103 - 207	Acceptable
Ethylbenzene	µg/L	11.7	10.0	5.45 - 14.8	Acceptable
Toluene	µg/L	80.6	84.8	53.9 - 112	Acceptable
Total Xylenes	µg/L	264	408	234 - 540	Acceptable
t-Butylmethylether (MTBE)	µg/L	132	162	79.9 - 204	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

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QuiK™ Response PE Standards

Final Report

BTEX in Water

Customer: Analytical Management Labs
Lot Number: 032905A-2
State ID Number: KS00902
Method: SW 8260B

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
Benzene	µg/L	149	163	103 - 207	Acceptable
Ethylbenzene	µg/L	11.1	10.0	5.45 - 14.8	Acceptable
Toluene	µg/L	88.0	84.8	53.9 - 112	Acceptable
Total Xylenes	µg/L	381	408	234 - 540	Acceptable
t-Butylmethylether (MTBE)	µg/L	131	162	79.9 - 204	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

ENVIRONMENTAL RESOURCE ASSOCIATES®
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QuiK™ Response PE Standards

Final Report

BTEX in Soil

Customer: Analytical Management Labs
Lot Number: 032905A-1
State ID Number: KS00902
Method: SW 8260B

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
Benzene	µg/kg	332	353	215 - 493	Acceptable
Ethylbenzene	µg/kg	172	152	75.3 - 248	Acceptable
Toluene	µg/kg	174	152	89.5 - 231	Acceptable
Total Xylenes	µg/kg	856	788	389 - 1130	Acceptable
t-Butylmethylether (MTBE)	µg/kg	269	328	173 - 409	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

QuiK™ Response PE Standards

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

Final Report

BTEX in Soil

Customer: Analytical Management Labs
Lot Number: 032905A-1
State ID Number: KS00902
Method: OK-GRO-8021B

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
Benzene	µg/kg	306	353	215 - 498	Acceptable
Ethylbenzene	µg/kg	130	152	75.3 - 243	Acceptable
Toluene	µg/kg	133	152	89.5 - 231	Acceptable
Total Xylenes	µg/kg	679	788	389 - 1130	Acceptable
t-Butylmethylether (MTBE)	µg/kg	262	328	173 - 409	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

ENVIRONMENTAL RESOURCE ASSOCIATES®
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QuiK™ Response PE Standards

Final Report

BTEX in Soil

Customer: Analytical Management Labs
Lot Number: 082905A-2
State ID Number: KS00902
Method: SW 8260B

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
Benzene	µg/kg	329	358	170 - 518	Acceptable
Ethylbenzene	µg/kg	163	151	77.0 - 215	Acceptable
Toluene	µg/kg	77.7	76.8	38.5 - 115	Acceptable
Total Xylenes	µg/kg	547	523	202 - 788	Acceptable
t-Butylmethylether (MTBE)	µg/kg	209	267	86.1 - 376	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

QuiK™ Response PE Standards

Final Report

BTEX in Soil

Customer: Analytical Management Labs
Lot Number: 032905A-2
State ID Number: KS00902
Method: OK-GRO-8021B

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
Benzene	µg/kg	302	358	170 - 518	Acceptable
Ethylbenzene	µg/kg	119	151	77.0 - 215	Acceptable
Toluene	µg/kg	65.6	76.8	38.5 - 115	Acceptable
Total Xylenes	µg/kg	427	523	202 - 788	Acceptable
t-Butylmethylether (MTBE)	µg/kg	200	267	86.1 - 376	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

ENVIRONMENTAL RESOURCE ASSOCIATES®
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QuiK™ Response PE Standards

Final Report

Diesel in Water

Customer: Analytical Management Labs
Lot Number: 032905A-1
State ID Number: KS00902
Method: SW 8015C

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
No. 2 Diesel	µg/L	1380	1990	0.00 - 2820	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

QuiK™ Response PE Standards

Final Report

Diesel in Water

Customer: Analytical Management Labs
Lot Number: 032905A-1
State ID Number: KS00902
Method: OK-DEQ-DRO-8100

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
No. 2 Diesel	µg/L	1760	1990	0.00 - 2820	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

QuiK™ Response PE Standards

Final Report

Diesel in Water

Customer: Analytical Management Labs
Lot Number: 032905A-2
State ID Number: KS00902
Method: SW 8015C

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
No. 2 Diesel	µg/L	540	887	24.0 - 1190	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

QuiK™ Response PE Standards

Final Report

Diesel in Water

Customer: Analytical Management Labs
Lot Number: 032905A-2
State ID Number: KS00902
Method: OK-DEQ-DRO-8100

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
No. 2 Diesel	µg/L	690	887	24.0 - 1190	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

QuiK™ Response PE Standards

Final Report

Diesel in Soil

Customer: Analytical Management Labs
Lot Number: 032905A-1
State ID Number: KS00902
Method: SW 8015C

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
No. 2 Diesel	mg/kg	745	848	254 - 1120	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

QuiK™ Response PE Standards

Final Report

Diesel in Soil

Customer: Analytical Management Labs
Lot Number: 032905A-1
State ID Number: KS00902
Method: OK-DEQ-DRO-8100

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
No. 2 Diesel	mg/kg	947	848	254 - 1120	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

ENVIRONMENTAL RESOURCE ASSOCIATES®
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QuiK™ Response PE Standards

Final Report

Diesel in Soil

Customer: Analytical Management Labs
Lot Number: 032905A-2
State ID Number: KS00902
Method: SW 8015C

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
No. 2 Diesel	mg/kg	1750	2080	599 - 2740	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

QuiK™ Response PE Standards

Final Report

Diesel in Soil

Customer: Analytical Management Labs
Lot Number: 032905A-2
State ID Number: KS00902
Method: OK-DEQ-DRO-8100

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Performance Evaluation
No. 2 Diesel	mg/kg	2220	2080	599 - 2740	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Results Received by ERA : 4/6/2005

Report Issue Date: 4/7/2005

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

Fax

To:	TENKASI VISWANATHAN	**From:**	Shawn Kassner
Company:	ANALYTICAL MANAGEMENT LABS	**Date:**	November 23, 2004
Fax:	913-829-1191	**Pages:**	5
Re:	Quick Response 102604H		

☐ **Urgent** ☒ **For Review** ☐ **Please Comment** ☐ **Please Reply** ☐ **Please Recycle**

Please feel free to call me if you have any questions.

Thanks,

Shawn

6000 West 54th Ave., Arvada, CO 80002 800-372-0122 fax (303) 421-0159 www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES
The Industry Standard™

QuiK™ Response PE Standards

Final Report

Unleaded Gasoline in Soil

Customer: Analytical Management Labs
Lot Number: 102604H-1
State ID Number: KS00902(EPA)/88-0873(AR)/E-10254(KS)
Method: 8015(Gasoline)/ 8260 (BTEX)

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Comment
Unleaded Gasoline	mg/kg	632	691	121 - 1080	Acceptable
Benzene	mg/kg	5.16	6.91	0.00 - 9.01	Acceptable
Ethylbenzene	mg/kg	15.5	17.3	4.60 - 23.2	Acceptable
Toluene	mg/kg	43.4	60.7	13.3 - 69.5	Acceptable
Xylenes, total	mg/kg	81.8	96.7	36.8 - 117	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Date of Report: 11/23/2004

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

QuiK™ Response PE Standards

Final Report

Unleaded Gasoline in Soil

Customer: Analytical Management Labs
Lot Number: 102604H-2
State ID Number: KS00902(EPA)/88-0873(AR)/E-10254(KS)
Method: 8015(Gasoline)/ 8260 (BTEX)

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Comment
Unleaded Gasoline	mg/kg	707	1110	289 - 1520	Acceptable
Benzene	mg/kg	8.11	11.1	0.00 - 11.4	Acceptable
Ethylbenzene	mg/kg	28.4	27.9	5.29 - 34.5	Acceptable
Toluene	mg/kg	73.2	97.5	16.7 - 100	Acceptable
Xylenes, total	mg/kg	152	155	41.6 - 178	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Date of Report: 11/23/2004

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

QuiK™ Response PE Standards

Final Report

No. 2 Diesel in Soil

Customer: Analytical Management Labs
Lot Number: 102604H-1
State ID Number: KS00902(EPA)/88-0873(AR)/E-10254(KS)
Method: EPA 8015

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Comment
No. 2 Diesel	mg/kg	641	897	252 - 1110	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Date of Report: 11/23/2004

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

QuiK™ Response PE Standards

Final Report

No. 2 Diesel in Soil

Customer: Analytical Management Labs
Lot Number: 102604H-2
State ID Number: KS00902(EPA)/88-0873(AR)/E-10254(KS)
Method: EPA 8015

Parameter	Units	Reported Value	Assigned Value	QuiK™ Response Limits	Comment
No. 2 Diesel	mg/kg	2410	3500	1370 - 4550	Acceptable

Results reported by: Tenkasi Viswanathan - Analytical Management Labs

Date of Report: 11/23/2004

ENVIRONMENTAL RESOURCE ASSOCIATES®

UST-48 Definitions & Study Discussion

ERA Laboratory Code: A5230-01 **EPA ID: KS00902**

Report Issued: 12/17/04
Study Dates: 10/15/04 - 11/29/04

UST Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Values are established per the National Environmental Laboratory Accreditation Conference (NELAC) program criteria.

The Acceptance Limits are established per the USEPA's guidelines contained in the National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, the NELAC PT program criteria, state specific PT program requirements or ERA's SOP for the Generation of Performance Acceptance Limits™ as applicable. For a further discussion of how your results were evaluated, please see the cover letter that accompanied the enclosed report.

The Performance Evaluation:

Acceptable = Reported Value falls within the Acceptance Limits.

Not Acceptable = Reported Value falls outside of the Acceptance Limits.

No Evaluation = Reported Value cannot be evaluated.

The Method Description is the method the laboratory reported to ERA.

UST Study Discussion:

ERA's Underground Storage Tank Proficiency Testing Study, UST-48, has been reviewed by ERA Senior Management and certified compliant with the requirements of the National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program, Chapter 2, July 2003.

Per the requirements of the NELAC Proficiency Testing Program, the UST-48 results were examined for any study anomalies. Based upon ERA's review of the data, no study anomalies were found.

A full review of all accuracy verification, homogeneity and stability (VHS) data was also completed. All analytical verification data for all analytes in the UST-48 standards met the acceptance criteria contained in the NELAC Proficiency Testing Program, Chapter 2, July 2003. All Acceptance Limits for the UST-48 Study were calculated based on the NELAC Proficiency Testing Program, Chapter 2, July 2003, as applicable.

The Underground Storage Tank Proficiency Testing Study UST-48 report shall not be reproduced except in its entirety and without the full written permission of the participating laboratory. The report must not be used by the participating laboratory to claim product endorsement by NVLAP or any agency of the U.S. government.

If you have any questions regarding the UST-48 Study, please contact Shawn Kassner, Proficiency Testing Manager, or Curtis Wood, Quality Assurance Director, at 1-800-372-0122.

NVLAP® Lab code: 200386-0

ENVIRONMENTAL RESOURCE ASSOCIATES®

Study: **UST-48**

ERA Laboratory Code: **A5230-01**

Laboratory Name: **Analytical Management Labs**

Report Type: **Complete**

Report Method: **Method A**

ACCREDITED Lab Code: 1539-01

Proficiency Testing Studies / Quality Control Standards

NVLAP Lab Code: 200386-0

6000 West 54th Ave., Arvada, CO 80002 1-800-372-0122 fax 303-421-0159 info@eraqc.com www.eraqc.com

ENVIRONMENTAL RESOURCE ASSOCIATES®

UST-48 Final Complete Report

Tenkasi S. Viswanathan
QA Director
Analytical Management Labs
15130 S Keeler
Suite B
Olathe, KS 66062

EPA ID: KS00902
ERA Laboratory Code: A5230-01
Report Issued: 12/17/04
Study Dates: 10/15/04 - 11/29/04

Anal. No.	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Gasoline in Soil							
9408	Unleaded Gasoline	mg/kg	127	357	0.00 - 456	Acceptable	EPA 8015B
4375	Benzene in Gas	mg/kg	1.90	3.57	0.00 - 3.57	Acceptable	EPA 8260B
4765	Ethylbenzene in Gas	mg/kg	5.13	8.96	0.00 - 11.2	Acceptable	EPA 8260B
5140	Toluene in Gas	mg/kg	10.1	31.3	0.00 - 33.1	Acceptable	EPA 8260B
5260	Xylenes, total in Gas	mg/kg	19.0	50.0	0.00 - 60.6	Acceptable	EPA 8260B
Diesel in Soil							
9369	No. 2 Diesel	mg/kg	320	395	120 - 520	Acceptable	EPA 8015B

All analytes are included in ERA's A2LA accreditation. Lab Code: 1539-01
† Indicates analytes included in ERA's NIST/NVLAP accreditation. Lab Code 200386-0

NVLAP® Labcode: 200386-0

AML Statement of Laboratory

Self Declaration of Compliance

AML Kansas Certification

Analytical Management Laboratories, Inc.
est. 1993

15130 South Keeler, Olathe, Kansas 66062
Phone: (913) 829-0101 • Fax: (913) 829-1181

United States Army Corps of Engineers
Candidate Environmental Laboratory
Self Declaration Form

Legal Name of Laboratory:	**Analytical Management Laboratories, Inc. (AML)**
Street Address:	15130 South Keeler Olathe, KS 66062
Name of Owner:	AML
Owner Address (if different)	Same as above
Phone Number:	913-829-0101
Fax Number:	913-829-1181
Website:	www.amlabinc.com
Email Address:	tviswanathan@amlabinc.com
Laboratory Director:	Bill Said
Phone Number:	913-829-0101, ext.25
Email Address:	bsaid@amlabinc.com
Quality Assurance Officer:	Tenkasi S. Viswanathan
Phone Number:	913-829-0101 ext.26
Email Address:	tviswanathan@amlabinc.com

The undersigned persons understand and acknowledge that:

1. To the best of our knowledge, laboratory operations that will be utilized for testing in support of environmental analytical testing for United States Army Corps of Engineers (USACE) are in full compliance with the most recent version of the Department of Defense (DoD) Quality Systems Manual (QSM), which includes national environmental laboratory accreditation conference (NELAC) standard chapter 5 and Appendix requirements.
2. All written documentation provided to USACE, accompanying this declaration accurately reflects policy and practices either already implemented or soon to be implemented by laboratory staff when recent revisions have been made to the referenced documents.
3. The laboratory will notify USACE immediately of change in status of laboratory operations that may affect on-going compliance as declared by items 1 and 2.

Multi State Certified

Analytical Management Laboratories, Inc.
est. 1993

15130 South Keeler, Olathe, Kansas 66062
Phone: (913) 829-0101 • Fax: (913) 829-1181

4. The laboratory acknowledges that USACE may audit the laboratory, relative to policy compliance at any time deemed appropriate; and will allow a designated Contracting Officer Representative (COR) full access to information and facilities to conduct such audit operations.
5. The laboratory will provide the following documents to the laboratory evaluator to very compliance with the QSM:
   a. A copy of the laboratory's most recent Quality Assurance Manual.
   b. A copy of the laboratory's ethics program policies and quality system procedures, which may include selected quality assurance (QA) standard operating procedures (SOPs) on method detection limit (MDL) studies and laboratory control samples (LCS) control chart limits.
   c. A copy of the determinative and preparatory method SOPs for the parameters for which testing will be performed.
   d. A copy of the method performance demonstration for the parameters of interest, which may include MDL studies, LCS control ranges, and proficiency testing (PT) sample results from a national environmental laboratory accreditation program (NELAP) accredited PT sample provider for the parameters of interest.
   e. A copy of the NELAC accreditation for the parameters of interest, when NELAC accreditation is offered is offered for the parameters.
6. Signatories are authorized to sign this form on behalf of the owner and to the best of our knowledge there are no misrepresentations in the information that will be provided in the laboratory assessment package.

Signature of Quality Assurance Officer:	
Date:	5/25/06
Signature of Laboratory Director:	
Date:	5/25/06

Multi State Certified

STATE OF KANSAS

DEPARTMENT OF HEALTH AND ENVIRONMENT

CERTIFICATE

This is to certify that Certificate No. E-10254

Analytical Management Laboratories Inc
15130 South Keeler Suite B
Olathe, KS 66062-2760

has been accredited in accordance with K.S.A. 65-1,109a for performing environmental analyses for the parameters listed on the attached form. Continuous accreditation depends on successful, ongoing participation in the program. Clients are urged to verify with this agency the laboratory's certification status for particular methods and analytes.

EFFECTIVE DATE: 05/01/2006 EXPIRATION DATE: 04/30/2007

Secretary
Department of Health and Environment

Environmental Laboratory Certification Officers

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KANSAS DEPARTMENT OF HEALTH AND ENVIRONMENT
ENVIRONMENTAL LABORATORY CERTIFICATION
DRINKING WATER CERTIFICATION - PARAMETER LIST

This certificate supersedes all previous certificates

Analytical Management Laboratories Inc
15130 South Keeler Suite B
Olathe, KS 66062-2760

Certificate Number: E-10254
Effective Date: 05/01/2006
Expiration Date: 04/30/2007
Reciprocity:

The laboratory listed above is hereby approved for environmental laboratory certification in accordance with K.S.A. 65-1,109a for performing drinking water analysis for the following parameters:

****METALS**
Aluminum {EPA 200.7}
Aluminum {EPA 200.8}
Antimony {EPA 200.8}
Arsenic {EPA 200.8}
Barium {EPA 200.7}
Barium {EPA 200.8}
Beryllium {EPA 200.7}
Beryllium {EPA 200.8}
Cadmium {EPA 200.7}
Cadmium {EPA 200.8}
Chromium {EPA 200.7}
Chromium {EPA 200.8}
Copper {EPA 200.7}
Copper {EPA 200.8}
Iron {EPA 200.7}
Lead {EPA 200.8}
Manganese {EPA 200.7}
Manganese {EPA 200.8}
Mercury {EPA 200.8}
Mercury {EPA 245.1}
Nickel {EPA 200.7}
Nickel {EPA 200.8}
Selenium {EPA 200.8}
Silver {EPA 200.7}
Silver {EPA 200.8}
Zinc {EPA 200.7}
Zinc {EPA 200.8}

****MINERALS**
Chloride {EPA 300.0}
Fluoride {EPA 300.0}
Sulfate {EPA 300.0}

NELAP-Recognized

****NUTRIENTS**
Nitrate {EPA 300.0}
Nitrate {EPA 353.2}
Nitrate {SM 4500-NO3 E}

****ORGANIC CHEMISTRY ADIPATE/PHTALATE**
{EPA 525.2} Di(ethylhexyl)adipate
{EPA 525.2} Di(ethylhexyl)phthalate

****ORGANIC CHEMISTRY DBCP/EDB**
{EPA 504.1} 1,2-Dibromo-3-Chloropropane (DBCP)
{EPA 504.1} 1,2-Dibromoethane (EDB, Ethylene dibromide)
{EPA 504.1} 1,2,3-Trichloropropane

****ORGANIC CHEMISTRY P.A.Hs**
{EPA 525.2} Benzo(a)pyrene

**ORGANIC CHEMISTRY VOLATILES
{EPA 524.2} 1,1-Dichloroethylene
{EPA 524.2} 1,1,1-Trichloroethane
{EPA 524.2} 1,1,2-Trichloroethane
{EPA 524.2} 1,2-Dichlorobenzene
{EPA 524.2} 1,2-Dichloroethane
{EPA 524.2} 1,2-Dichloropropane
{EPA 524.2} 1,2,4-Trichlorobenzene
{EPA 524.2} 1,4-Dichlorobenzene
{EPA 524.2} Benzene
{EPA 524.2} Carbon Tetrachloride
{EPA 524.2} Chlorobenzene
{EPA 524.2} cis-1,2-Dichloroethylene
{EPA 524.2} Dichloromethane (Methylene chloride)
{EPA 524.2} Ethylbenzene
{EPA 524.2} Styrene
{EPA 524.2} Tetrachloroethylene
{EPA 524.2} Toluene
{EPA 524.2} trans-1,2-Dichloroethylene
{EPA 524.2} Trichloroethylene
{EPA 524.2} Vinyl Chloride
{EPA 524.2} Xylene

**RESIDUES
Residue, Filterable (TDS) {SM 2540C}

**TRIHALOMETHANES
{EPA 524.2} Bromodichloromethane
{EPA 524.2} Bromoform
{EPA 524.2} Chloroform
{EPA 524.2} Dibromochloromethane

**UNREGULATED ORGANIC COMPOUNDS
{EPA 524.2} 1,1-Dichloroethane
{EPA 524.2} 1,1-Dichloropropene
{EPA 524.2} 1,1,1,2-Tetrachloroethane
{EPA 524.2} 1,1,2,2-Tetrachloroethane
{EPA 524.2} 1,2,3-Trichlorobenzene
{EPA 524.2} 1,2,3-Trichloropropane
{EPA 524.2} 1,2,4-Trimethylbenzene
{EPA 524.2} 1,3-Dichlorobenzene
{EPA 524.2} 1,3-Dichloropropane
{EPA 524.2} 1,3,5-Trimethylbenzene
{EPA 524.2} 2-Chlorotoluene
{EPA 524.2} 2,2-Dichloropropane
{EPA 524.2} 4-Chlorotoluene
{EPA 524.2} 4-Isopropyltoluene
{EPA 524.2} Bromobenzene
{EPA 524.2} Bromochloromethane
{EPA 524.2} Bromomethane
{EPA 524.2} Chloroethane
{EPA 524.2} Chloromethane
{EPA 524.2} cis-1,3-Dichloropropene
{EPA 524.2} Dibromomethane
{EPA 524.2} Dichlorodifluoromethane
{EPA 524.2} Hexachlorobutadiene
{EPA 524.2} Isopropylbenzene
{EPA 524.2} Methyl-t-butyl ether
{EPA 524.2} n-Butylbenzene
{EPA 524.2} n-Propylbenzene

NELAP-Recognized

{EPA 524.2} Naphthalene
{EPA 524.2} sec-Butylbenzene
{EPA 524.2} tert-Butylbenzene
{EPA 524.2} trans-1,3-Dichloropropene
{EPA 524.2} Trichlorofluoromethane

End of Parameter List

NELAP-Recognized

KANSAS DEPARTMENT OF HEALTH AND ENVIRONMENT
ENVIRONMENTAL LABORATORY CERTIFICATION
WASTE WATER CERTIFICATION - PARAMETER LIST

This certificate supersedes all previous certificates

Analytical Management Laboratories Inc	**Certificate Number: E-10254**
15130 South Keeler Suite B	**Effective Date: 05/01/2006**
Olathe, KS 66062-2760	**Expiration Date: 04/30/2007**
	Reciprocity:

The laboratory listed above is hereby approved for environmental laboratory certification in accordance with K.S.A. 65-1,109a for performing waste water analysis for the following parameters:

****DEMANDS**
BOD {EPA 405.1}
BOD {SM 5210B}
cBOD {SM 5210B}
Oxygen, Dissolved {EPA 360.1}

****INORGANIC**
Chlorine - total {EPA 330.4}
Cyanide, Amenable to CL {EPA 335.1}
Cyanide, Amenable to CL {SM 4500-CN-G}

****METALS**
Aluminum {EPA 200.7}
Aluminum {EPA 200.8 - ATP}
Antimony {EPA 200.7}
Antimony {EPA 200.8 - ATP}
Arsenic {EPA 200.7}
Arsenic {EPA 200.8 - ATP}
Barium {EPA 200.7}
Barium {EPA 200.8 - ATP}
Beryllium {EPA 200.7}
Beryllium {EPA 200.8 - ATP}
Boron {EPA 200.7}
Cadmium {EPA 200.7}
Cadmium {EPA 200.8 - ATP}
Calcium {EPA 200.7}
Chromium {EPA 200.7}
Chromium {EPA 200.8 - ATP}
Cobalt {EPA 200.7}
Cobalt {EPA 200.8 - ATP}
Copper {EPA 200.7}
Copper {EPA 200.8 - ATP}
Iron {EPA 200.7}
Lead {EPA 200.7}
Lead {EPA 200.8 - ATP}
Magnesium {EPA 200.7}
Manganese {EPA 200.7}
Manganese {EPA 200.8 - ATP}
Mercury {EPA 245.1}
Molybdenum {EPA 200.7}
Molybdenum {EPA 200.8 - ATP}
Nickel {EPA 200.7}
Nickel {EPA 200.8 - ATP}
Potassium {EPA 200.7}
Selenium {EPA 200.7}
Selenium {EPA 200.8 - ATP}
Silver {EPA 200.7}
Silver {EPA 200.8 - ATP}
Sodium {EPA 200.7}
Thallium {EPA 200.7}

NELAP-Recognized

Thallium {EPA 200.8 - ATP}
Tin {EPA 200.7}
Tin {EPA 200.8 - ATP}
Titanium {EPA 200.7-ATP}
Vanadium {EPA 200.7}
Vanadium {EPA 200.8 - ATP}
Zinc {EPA 200.7}
Zinc {EPA 200.8 - ATP}

**METALS - 503 Regs
Arsenic {EPA 6010B}
Arsenic {EPA 6020}
Cadmium {EPA 6010B}
Cadmium {EPA 6020}
Chromium, Total {EPA 6010B}
Chromium, Total {EPA 6020}
Copper {EPA 6010B}
Copper {EPA 6020}
Lead {EPA 6010B}
Lead {EPA 6020}
Mercury {EPA 6020}
Mercury {EPA 7470}
Mercury {EPA 7471}
Molybdenum {EPA 6010B}
Molybdenum {EPA 6020}
Nickel {EPA 6010B}
Nickel {EPA 6020}
Selenium {EPA 6010B}
Selenium {EPA 6020}
Zinc {EPA 6010B}
Zinc {EPA 6020}

**MINERALS
Acidity {EPA 305.1}
Acidity {SM 2310B}
Alkalinity {EPA 310.1}
Alkalinity {SM 2320B}
Chloride {EPA 300.0}
Chloride {EPA 325.3}
Fluoride {EPA 300.0}
Hardness {SM 2340B}
Sulfate {EPA 300.0}
Sulfate {EPA 375.4}
Sulfite {EPA 377.1}
Sulfite {SM 4500-SO3-2 B}

NELAP-Recognized

**MISCELLANEOUS
Bromide {EPA 300.0}
Color {EPA 110.2}
Hydrogen Ion (pH) {EPA 150.1}
Oil & Grease {EPA 1664}
Oil & Grease {SM 5520B}
Temperature {EPA 170.1}
Turbidity {EPA 180.1}

**NUTRIENTS
Ammonia {EPA 350.3}
Nitrate {EPA 300.0}
Nitrate {EPA 353.3}
Nitrite {EPA 300.0}
Nitrite {EPA 354.1}

O-Phosphate {EPA 300.0}
O-Phosphate {EPA 365.3}
Phosphorus {EPA 365.3}
Phosphorus {SM 4500-P E}

**ORGANIC CHEMISTRY VOLATILES (MEASUREMENT BY GC/MS)
{EPA 624} 1,1-Dichloroethane
{EPA 624} 1,1-Dichloroethylene
{EPA 624} 1,1,1-Trichloroethane
{EPA 624} 1,1,2-Trichloroethane
{EPA 624} 1,1,2,2-Tetrachloroethane
{EPA 624} 1,2-Dichlorobenzene
{EPA 624} 1,2-Dichloroethane
{EPA 624} 1,2-Dichloropropane
{EPA 624} 1,3-Dichlorobenzene
{EPA 624} 1,4-Dichlorobenzene
{EPA 624} 2-Chloroethyl vinyl ether
{EPA 624} Acrolein
{EPA 624} Acrylonitrile
{EPA 624} Benzene
{EPA 624} Bromodichloromethane
{EPA 624} Bromoform
{EPA 624} Bromomethane
{EPA 624} Carbon Tetrachloride
{EPA 624} Chlorobenzene
{EPA 624} Chloroethane
{EPA 624} Chloroform
{EPA 624} Chloromethane
{EPA 624} cis-1,3-Dichloropropene
{EPA 624} Dibromochloromethane
{EPA 624} Dichloromethane (Methylene chloride)
{EPA 624} Ethylbenzene
{EPA 624} Tetrachloroethylene
{EPA 624} Toluene
{EPA 624} trans-1,2-Dichloroethylene
{EPA 624} trans-1,3-Dichloropropene
{EPA 624} Trichloroethylene
{EPA 624} Trichlorofluoromethane
{EPA 624} Vinyl Chloride

**ORGANIC CHEMISTRY (MEASUREMENT BY GC)
{EPA 608} 4,4'-DDD
{EPA 608} 4,4'-DDE
{EPA 608} 4,4'-DDT
{EPA 608} Aldrin
{EPA 608} alpha-BHC (alpha-Hexachlorocyclohexane)
{EPA 608} beta-BHC (beta-Hexachlorocyclohexane)
{EPA 608} Chlordane
{EPA 608} delta-BHC
{EPA 608} Dieldrin
{EPA 608} Endosulfan I
{EPA 608} Endosulfan II
{EPA 608} Endosulfan Sulfate
{EPA 608} Endrin
{EPA 608} Endrin aldehyde
{EPA 608} gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)
{EPA 608} Heptachlor
{EPA 608} Heptachlor epoxide
{EPA 608} PCB-1016
{EPA 608} PCB-1221
{EPA 608} PCB-1232

ACCREDITING AUTHORITY nelap
NELAP-Recognized

{EPA 608} PCB-1248
{EPA 608} PCB-1254
{EPA 608} PCB-1260
{EPA 608} Toxaphene (Chlorinated camphene)

**ORGANIC CHEMISTRY (MEASUREMENT BY GC/MS)
{EPA 625} 1,2-Dichlorobenzene
{EPA 625} 1,2,4-Trichlorobenzene
{EPA 625} 1,3-Dichlorobenzene
{EPA 625} 1,4-Dichlorobenzene
{EPA 625} 2-Chloronaphthalene
{EPA 625} 2-Chlorophenol
{EPA 625} 2-Methyl-4,6-Dinitrophenol
{EPA 625} 2-Nitrophenol
{EPA 625} 2,4-Dichlorophenol
{EPA 625} 2,4-Dimethylphenol
{EPA 625} 2,4-Dinitrophenol
{EPA 625} 2,4-Dinitrotoluene (2,4-DNT)
{EPA 625} 2,4,6-Trichlorophenol
{EPA 625} 2,6-Dinitrotoluene
{EPA 625} 3,3'-Dichlorobenzidine
{EPA 625} 4-Bromophenyl phenyl ether
{EPA 625} 4-Chloro-3-methylphenol
{EPA 625} 4-Chlorophenyl phenyl ether
{EPA 625} 4-Nitrophenol
{EPA 625} Acenaphthene
{EPA 625} Acenaphthylene
{EPA 625} Anthracene
{EPA 625} Benzidine
{EPA 625} Benzo(a)anthracene
{EPA 625} Benzo(a)pyrene
{EPA 625} Benzo(b)fluoranthene
{EPA 625} Benzo(g,h,i)perylene
{EPA 625} Benzo(k)fluoranthene
{EPA 625} Benzyl butyl phthalate
{EPA 625} Bis(2-chloroethoxy)methane
{EPA 625} Bis(2-chloroethyl)ether
{EPA 625} bis(2-Chloroisopropyl)ether
{EPA 625} Bis(2-ethylhexyl)phthalate
{EPA 625} Chrysene
{EPA 625} Di-n-butyl phthalate
{EPA 625} Di-n-octyl phthalate
{EPA 625} Dibenzo(a,h)anthracene
{EPA 625} Diethyl phthalate
{EPA 625} Dimethyl phthalate
{EPA 625} Fluoranthene
{EPA 625} Fluorene
{EPA 625} Hexachlorobenzene
{EPA 625} Hexachlorobutadiene
{EPA 625} Hexachlorocyclopentadiene
{EPA 625} Hexachloroethane
{EPA 625} Indeno(1,2,3-c,d)pyrene
{EPA 625} Isophorone
{EPA 625} N-Nitrosodi-n-propylamine
{EPA 625} N-Nitrosodimethylamine
{EPA 625} N-Nitrosodiphenylamine
{EPA 625} Naphthalene
{EPA 625} Nitrobenzene
{EPA 625} Pentachlorophenol
{EPA 625} Phenanthrene
{EPA 625} Phenol

ACCREDITING AUTHORITY
nelap
NELAP-Recognized

{EPA 625} Pyrene

****RESIDUES**
Residue, Filterable (TDS) {EPA 160.1}
Residue, Filterable (TDS) {SM 2540C}
Residue, Non Filterable (TSS) {EPA 160.2}
Residue, Non Filterable (TSS) {SM 2540D}
Residue, Settleable {EPA 160.5}
Residue, Settleable {SM 2540F}
Residue, Total {EPA 160.3}
Residue, Total {SM 2540B}
Residue, Volatile {EPA 160.4}

End of Parameter List

NELAP-Recognized

KANSAS DEPARTMENT OF HEALTH AND ENVIRONMENT
ENVIRONMENTAL LABORATORY CERTIFICATION
SOLID/HAZARDOUS WASTE CERTIFICATION - PARAMETER LIST

This certificate supersedes all previous certificates

Analytical Management Laboratories Inc	**Certificate Number: E-10254**
15130 South Keeler Suite B	**Effective Date: 05/01/2006**
Olathe, KS 66062-2760	**Expiration Date: 04/30/2007**
	Reciprocity:

The laboratory listed above is hereby approved for environmental laboratory certification in accordance with K.S.A. 65-1,109a for performing solids and/or hazardous waste analysis for the following parameters:

****CHARACTERISTICS**
Corrosivity {EPA 1110}
Extraction Procedure Toxicity {EPA 1310A}
Ignitability {EPA 1010}
Ignitability {EPA 1020A}
Mobile Metal Concentration in Oily Waste {EPA 1330A}
Multiple Extraction Procedure {EPA 1320}
Reactive Cyanide {CHAP 7, SEC. 7.3}
Reactive Sulfide {CHAP 7, SEC. 7.3}
Synthetic Precipitation Leaching Procedure {EPA 1312}
Toxic Characteristic Leaching Procedure {EPA 1311}

****DEMANDS**
Total Organic Carbon {EPA 9060}

****INORGANIC**
Cyanide, Amenable to CL {EPA 9010B}
Cyanide, Total {EPA 9010B}
Extractable Cyanide {EPA 9013}

****METALS**
Aluminum {EPA 6010B}
Aluminum {EPA 6020}
Antimony {EPA 6010B}
Antimony {EPA 6020}
Arsenic {EPA 6010B}
Arsenic {EPA 6020}
Barium {EPA 6010B}
Barium {EPA 6020}
Beryllium {EPA 6010B}
Beryllium {EPA 6020}
Boron {EPA 6010B}
Cadmium {EPA 6010B}
Cadmium {EPA 6020}
Calcium {EPA 6010B}
Chromium {EPA 6010B}
Chromium {EPA 6020}
Chromium, VI {EPA 7196A}
Cobalt {EPA 6010B}
Cobalt {EPA 6020}
Copper {EPA 6010B}
Copper {EPA 6020}
Iron {EPA 6010B}
Lead {EPA 6010B}
Lead {EPA 6020}
Magnesium {EPA 6010B}
Manganese {EPA 6010B}
Manganese {EPA 6020}
Mercury {EPA 7470A}

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Mercury {EPA 7471A}
Molybdenum {EPA 6010B}
Nickel {EPA 6010B}
Nickel {EPA 6020}
Potassium {EPA 6010B}
Selenium {EPA 6010B}
Silver {EPA 6010B}
Silver {EPA 6020}
Sodium {EPA 6010B}
Thallium {EPA 6010B}
Thallium {EPA 6020}
Vanadium {EPA 6010B}
Zinc {EPA 6010B}
Zinc {EPA 6020}

**MINERALS
Bromide {EPA 9056}
Chloride {EPA 9056}
Fluoride {EPA 9056}
Purgeable Sulfides {EPA 9031}
Sulfate {EPA 9035}
Sulfate {EPA 9056}
Sulfide {EPA 9030B}
Sulfide {EPA 9034}

**MISCELLANEOUS
Cation-Exchange Capacity of Soils {EPA 9080}
Cation-Exchange Capacity of Soils {EPA 9081}
Hydrogen Ion (pH) {EPA 9040B}
Hydrogen Ion (pH) {EPA 9041A}
Hydrogen Ion (pH) {EPA 9045C}
Liquid Release Test Procedure (LRT) {EPA 9096}
Oil & Grease {EPA 9070A}
Oil & Grease {EPA 9071B}
Paint Filter Liquids Test {EPA 9095A}
Phenolics {EPA 9065}
Specific Conductance {EPA 9050A}
Total Chlorine in New & Used Petroleum Products {EPA 9075}

**NUTRIENTS
Nitrate {EPA 9056}
Nitrite {EPA 9056}
Phosphate, Ortho {EPA 9056}

**ORGANIC CHEMISTRY VOLATILES (MEASUREMENT BY GC)
{EPA 8015B} Ethylene glycol

**ORGANIC CHEMISTRY VOLATILES (MEASUREMENT BY GC/MS)
{EPA 8260B} 1-Propanol
{EPA 8260B} 1,1-Dichloroethane
{EPA 8260B} 1,1-Dichloroethylene
{EPA 8260B} 1,1-Dichloropropene
{EPA 8260B} 1,1,1-Trichloroethane
{EPA 8260B} 1,1,1,2-Tetrachloroethane
{EPA 8260B} 1,1,2-Trichloroethane
{EPA 8260B} 1,1,2,2-Tetrachloroethane
{EPA 8260B} 1,2-Dibromoethane (EDB, Ethylene dibromide)
{EPA 8260B} 1,2-Dichlorobenzene
{EPA 8260B} 1,2-Dichloroethane
{EPA 8260B} 1,2-Dichloropropane
{EPA 8260B} 1,2,3-Trichlorobenzene

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{EPA 8260B} 1,2,3-Trichloropropane
{EPA 8260B} 1,2,4-Trichlorobenzene
{EPA 8260B} 1,2,4-Trimethylbenzene
{EPA 8260B} 1,3-Dichlorobenzene
{EPA 8260B} 1,3-Dichloropropane
{EPA 8260B} 1,3,5-Trimethylbenzene
{EPA 8260B} 1,4-Dichlorobenzene
{EPA 8260B} 1,4-Dioxane
{EPA 8260B} 2-Chloroethyl vinyl ether
{EPA 8260B} 2-Chlorotoluene
{EPA 8260B} 2-Hexanone
{EPA 8260B} 2-Pentanone
{EPA 8260B} 2-Propanol
{EPA 8260B} 2,2-Dichloropropane
{EPA 8260B} 4-Chlorotoluene
{EPA 8260B} 4-Isopropyltoluene
{EPA 8260B} 4-Methyl-2-Pentanone (MIBK)
{EPA 8260B} Acetone
{EPA 8260B} Acetonitrile
{EPA 8260B} Acrolein
{EPA 8260B} Acrylonitrile
{EPA 8260B} Allyl Chloride
{EPA 8260B} Benzene
{EPA 8260B} Bromobenzene
{EPA 8260B} Bromochloromethane
{EPA 8260B} Bromodichloromethane
{EPA 8260B} Bromoform
{EPA 8260B} Bromomethane
{EPA 8260B} Carbon disulfide
{EPA 8260B} Carbon Tetrachloride
{EPA 8260B} Chlorobenzene
{EPA 8260B} Chloroethane
{EPA 8260B} Chloroform
{EPA 8260B} Chloromethane
{EPA 8260B} cis-1,2-Dichloroethylene
{EPA 8260B} cis-1,3-Dichloropropene
{EPA 8260B} Dibromochloromethane
{EPA 8260B} Dibromofluoromethane
{EPA 8260B} Dibromomethane
{EPA 8260B} Dichlorodifluoromethane
{EPA 8260B} Dichloromethane (Methylene chloride)
{EPA 8260B} Diethyl ether
{EPA 8260B} Ethanol
{EPA 8260B} Ethyl acetate
{EPA 8260B} Ethyl methacrylate
{EPA 8260B} Ethylbenzene
{EPA 8260B} Hexachlorobutadiene
{EPA 8260B} Hexachloroethane
{EPA 8260B} Iodomethane
{EPA 8260B} Isobutyl Alcohol
{EPA 8260B} Isopropylbenzene
{EPA 8260B} meta-Xylene
{EPA 8260B} Methacrylonitrile
{EPA 8260B} Methyl acrylate
{EPA 8260B} Methyl ethyl ketone
{EPA 8260B} Methyl tert-butyl ether (MTBE)
{EPA 8260B} Methylmethacrylate
{EPA 8260B} n-Butyl alcohol
{EPA 8260B} n-Butylbenzene
{EPA 8260B} n-Propylbenzene
{EPA 8260B} Naphthalene

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{EPA 8260B} ortho-Xylene
{EPA 8260B} para-Xylene
{EPA 8260B} Propionitrile
{EPA 8260B} Pyridine
{EPA 8260B} sec-Butylbenzene
{EPA 8260B} Styrene
{EPA 8260B} t-Butyl alcohol
{EPA 8260B} tert-Butylbenzene
{EPA 8260B} Tetrachloroethylene
{EPA 8260B} Toluene
{EPA 8260B} trans-1,2-Dichloroethylene
{EPA 8260B} trans-1,3-Dichloropropene
{EPA 8260B} trans-1,4-Dichloro-2-butene
{EPA 8260B} Trichloroethylene
{EPA 8260B} Trichlorofluoromethane
{EPA 8260B} Vinyl Acetate
{EPA 8260B} Vinyl Chloride

**ORGANIC CHEMISTRY (MEASUREMENT BY GC)
{EPA 8081A} 4,4'-DDD
{EPA 8081A} 4,4'-DDE
{EPA 8081A} 4,4'-DDT
{EPA 8081A} Alachlor
{EPA 8081A} Aldrin
{EPA 8081A} alpha-BHC (alpha-Hexachlorocyclohexane)
{EPA 8081A} alpha-Chlordane
{EPA 8081A} beta-BHC (beta-Hexachlorocyclohexane)
{EPA 8081A} Chlordane (Tech)
{EPA 8081A} delta-BHC
{EPA 8081A} Dieldrin
{EPA 8081A} Endosulfan I
{EPA 8081A} Endosulfan II
{EPA 8081A} Endosulfan Sulfate
{EPA 8081A} Endrin
{EPA 8081A} Endrin aldehyde
{EPA 8081A} Endrin ketone
{EPA 8081A} g-Chlordane
{EPA 8081A} Heptachlor
{EPA 8081A} Heptachlor Epoxide
{EPA 8081A} Methoxychlor
{EPA 8081A} Mirex
{EPA 8081A} Propachlor (Ramrod)
{EPA 8081A} Toxaphene (Chlorinated camphene)
{EPA 8081A} trans-Nonachlor
{EPA 8081A} Trifluralin (Treflan)
{EPA 8082} PCB-1016
{EPA 8082} PCB-1221
{EPA 8082} PCB-1232
{EPA 8082} PCB-1242
{EPA 8082} PCB-1248
{EPA 8082} PCB-1254
{EPA 8082} PCB-1260
{EPA 8151A} 2,4-DB
{EPA 8151A} 2,4,5-T
{EPA 8151A} 2,4,5-TP
{EPA 8151A} 3,5-Dichlorobenzoic acid
{EPA 8151A} 4-Nitrophenol
{EPA 8151A} 5-Hydroxydicamba
{EPA 8151A} Aciflurofen
{EPA 8151A} Bentazon
{EPA 8151A} Chloramben

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{EPA 8151A} Dalapon
{EPA 8151A} DCPA Di-Acid
{EPA 8151A} Dicamba
{EPA 8151A} Dichlorprop
{EPA 8151A} Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)
{EPA 8151A} MCPA
{EPA 8151A} Pentachlorophenol
{EPA 8151A} Picloram

**ORGANIC CHEMISTRY (MEASUREMENT BY GC/MS)
{EPA 8270C} 1-Chloronaphthalene
{EPA 8270C} 1,2-Dichlorobenzene
{EPA 8270C} 1,2-Dinitrobenzene
{EPA 8270C} 1,2-Diphenylhydrazine
{EPA 8270C} 1,2,4-Trichlorobenzene
{EPA 8270C} 1,2,4,5-Tetrachlorobenzene
{EPA 8270C} 1,3-Dichlorobenzene
{EPA 8270C} 1,3,5-Trinitrobenzene
{EPA 8270C} 1,4-Dichlorobenzene
{EPA 8270C} 2-Chloronaphthalene
{EPA 8270C} 2-Chlorophenol
{EPA 8270C} 2-Methyl-4,6-Dinitrophenol
{EPA 8270C} 2-Methylnaphthalene
{EPA 8270C} 2-Methylphenol (o-Cresol)
{EPA 8270C} 2-Naphthylamine
{EPA 8270C} 2-Nitroaniline
{EPA 8270C} 2-Nitrophenol
{EPA 8270C} 2,3,4,6-Tetrachlorophenol
{EPA 8270C} 2,4-Dichlorophenol
{EPA 8270C} 2,4-Dimethylphenol
{EPA 8270C} 2,4-Dinitrophenol
{EPA 8270C} 2,4-Dinitrotoluene (2,4-DNT)
{EPA 8270C} 2,4,5-Trichlorophenol
{EPA 8270C} 2,4,6-Trichlorophenol
{EPA 8270C} 2,6-Dichlorophenol
{EPA 8270C} 2,6-Dinitrotoluene
{EPA 8270C} 3-Methylphenol (m-Cresol)
{EPA 8270C} 3-Nitroaniline
{EPA 8270C} 3,3'-Dichlorobenzidine
{EPA 8270C} 3,3'-Dimethylbenzidine
{EPA 8270C} 4-Bromophenyl phenyl ether
{EPA 8270C} 4-Chloro-3-methylphenol
{EPA 8270C} 4-Chloroaniline
{EPA 8270C} 4-Chlorophenyl phenyl ether
{EPA 8270C} 4-Methylphenol (p-Cresol)
{EPA 8270C} 4-Nitroaniline
{EPA 8270C} 4-Nitrophenol
{EPA 8270C} Acenaphthene
{EPA 8270C} Acenaphthylene
{EPA 8270C} Acetophenone
{EPA 8270C} Aniline
{EPA 8270C} Anthracene
{EPA 8270C} Benzidine
{EPA 8270C} Benzoic acid
{EPA 8270C} Benzo(a)anthracene
{EPA 8270C} Benzo(a)pyrene
{EPA 8270C} Benzo(b)fluoranthene
{EPA 8270C} Benzo(g,h,i)perylene
{EPA 8270C} Benzo(k)fluoranthene
{EPA 8270C} Benzyl alcohol
{EPA 8270C} Bis(2-chloroethoxy)methane

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{EPA 8270C} Bis(2-chloroethyl)ether
{EPA 8270C} Bis(2-chloroisopropyl)ether
{EPA 8270C} Bis(2-Ethylhexyl) Phthalate
{EPA 8270C} Butyl benzyl phthalate
{EPA 8270C} Chlorobenzilate
{EPA 8270C} Chrysene
{EPA 8270C} Di-n-butyl phthalate
{EPA 8270C} Di-n-octyl phthalate
{EPA 8270C} Dibenzofuran
{EPA 8270C} Dibenzo(a,h)anthracene
{EPA 8270C} Diethyl phthalate
{EPA 8270C} Dimethyl phthalate
{EPA 8270C} Diphenylamine
{EPA 8270C} Ethyl methanesulfonate
{EPA 8270C} Fluoranthene
{EPA 8270C} Fluorene
{EPA 8270C} Hexachlorobenzene
{EPA 8270C} Hexachlorobutadiene
{EPA 8270C} Hexachlorocyclopentadiene
{EPA 8270C} Hexachloroethane
{EPA 8270C} Indeno(1,2,3-c,d)pyrene
{EPA 8270C} Isophorone
{EPA 8270C} Methylmethanesulfonate
{EPA 8270C} N-Nitrosodi-n-butylamine
{EPA 8270C} N-Nitrosodi-n-propylamine
{EPA 8270C} N-Nitrosodiethylamine
{EPA 8270C} N-Nitrosodimethylamine
{EPA 8270C} N-Nitrosodiphenylamine
{EPA 8270C} Naphthalene
{EPA 8270C} Nitrobenzene
{EPA 8270C} Pentachlorobenzene
{EPA 8270C} Pentachlorophenol
{EPA 8270C} Phenacetin
{EPA 8270C} Phenanthrene
{EPA 8270C} Phenol
{EPA 8270C} Pyrene
{EPA 8270C} Pyridine

**ORGANIC CHEMISTRY (MEASUREMENT BY HPLC)
{EPA 8310} Acenaphthene
{EPA 8310} Acenaphthylene
{EPA 8310} Anthracene
{EPA 8310} Benzo(a)anthracene
{EPA 8310} Benzo(a)pyrene
{EPA 8310} Benzo(b)fluoranthene
{EPA 8310} Benzo(g,h,i)perylene
{EPA 8310} Benzo(k)fluoranthene
{EPA 8310} Chrysene
{EPA 8310} Dibenzo(a,h)anthracene
{EPA 8310} Fluoranthene
{EPA 8310} Fluorene
{EPA 8310} Indeno(1,2,3-c,d)pyrene
{EPA 8310} Naphthalene
{EPA 8310} Phenanthrene
{EPA 8310} Pyrene
{EPA 8330} 1,3-Dinitrobenzene
{EPA 8330} 1,3,5-Trinitrobenzene
{EPA 8330} 2-Amino-4,6-dinitrotoluene
{EPA 8330} 2,4-Dinitrotoluene (2,4-DNT)
{EPA 8330} 2,4,6-Trinitrotoluene
{EPA 8330} 2,6-Dinitrotoluene

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{EPA 8330} 4-Amino-2,6-dinitrotoluene
{EPA 8330} HMX
{EPA 8330} meta-Nitrotoluene
{EPA 8330} Methyl-2,4,6-trinitrophenylnitramine
{EPA 8330} Nitrobenzene
{EPA 8330} ortho-Nitrotoluene
{EPA 8330} para-Nitrotoluene
{EPA 8330} RDX
{EPA 8332} Nitroglycerine

****Total Petroleum Hydrocarbons**
{EPA 8015B} DRO
{EPA 8015B} GRO

End of Parameter List

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APPENDIX B
ENVIRONMENTAL PRIORITY SERVICE, INC.

Groundwater, Soil, and Soil Gas
Collection and On-Site Analysis
Utilizing Heated Headspace Method

Field Procedures Manual

ENVIRONMENTAL PRIORITY SERVICE, INC.

GROUNDWATER, SOIL & SOIL GAS
COLLECTION AND ON-SITE ANALYSIS
UTILIZING HEATED HEADSPACE METHOD

FIELD PROCEDURES MANUAL

August, 2006

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SAMPLING EQUIPMENT

EQUIPMENT

Probing Machine and Related Sampling Equipment

- Geoprobe 4200 & 5400 Series Hydraulic Probe Machines
- Geoprobe 6620 Track Unit
- On-board vacuum/volume system for vapor extraction
- Hardened alloy steel probe roads (1.25" x 3,4,5' flights)
- Larger diameter (2.125" & 3.25") probe rods for mini-well installation and track unit use
- Hollow Stem Augers for shallow (<30') 2" conventional wells
- Soil sampling tools: 4 & 5 ft. macros & 4 & 2 ft. discreet large-bore sampler
- Groundwater sampling tools: 2 & 4' mill-slotted, 4' stainless steel screen
- Groundwater screen implants
- Expendable Drive Points
- Polyethylene tubing for sampling
- Soil Gas sampling tools: PRT system
- Soil Electrical Conductivity System
- Grout Pump
- Generators
- Water Tanks
- Power Washers
- Holding tanks

Analytical Equipment

- Shimadzu model GC-14A laboratory grade gas chromatograph
- Hewlett Packard model 5890A Series 2 computerized laboratory gas chromatograph
- Shimadzu model CR-7A Chromatopac Data Processor for Chromatography
- P/E Nelson model 970A chromatography signal interface
- P/E Nelson Analytical Turbochrom 4.0 series integration software
- Three detectors – Flame Ionization (FID), Electron Capture (ECD), & Photoionization (PID)
- Temperature-controlled laboratory oven
- Odyssey Spectrophotometer

EQUIPMENT SPECIFICATIONS

Gas Chromatograph (GC-14A)

- Two temperature-controlled injector ports and three detectors (FID, ECD, & PID)
- Temperature Range from -80°C to +399°C
- Provides 5 Program Ramping Stages with a maximum temperature range of 40°C/minute
- Injection Port Range is 399°C

Data Integrator (CR-7A)

- Contains 2 imprint channels.
- Maximum peak number for identification: 1000 peaks
- Minimum peak width processed: 0.04 seconds (width at half-height)
- Linearity: less than 0.1%
- Dynamic Range: 10E6
- Disk Drives: 3.5" hard disk drive (40 Mbyte), 3.5" floppy disk drive (1.6 Mbyte)

Gas Chromatograph (HP-5890 Series 2)

- Two temperature-controlled injector ports and three detectors (FID, ECD, & PID)
- Temperature Range from -80°C to +450°C
- Connected in-line with Tekmar Purge and Trap
- Provides 3 Program Ramping Stages with a maximum temperature range of 40°C/minute
- Injection Port Range is 0-399°C

Signal Interface/Chromatography Integrator (Nelson model 970A)

- Contains 2 imprint channels.
- Maximum peak number for identification: 1000 peaks
- Minimum peak width processed: 0.04 seconds (width at half-height)
- Linearity: less than 0.1%

Megabore Column

- Maximum temperature: 260°C
- Type of Column: J&W DB-624
- Length of Column: 30 meters
- Outer Diameter: 0.543 mm

PRELIMINARY INSTRUMENT OPERATING CONDITIONS

The Hewlett Packard 5890A Series 2 Gas Chromatograph or the Shimadzu Gas Chromatograph 14-A (GC), each equipped with a photo-ionization detector (PID) and an electron capture detector (ECD) in series, will be utilized to analyze the samples. The GC column temperature will run isothermally for 3 minutes at 50° C, then temperature ramp at 10°C/min to 170°C, while the injector temperature is at 180° C and the detectors are at 225° C.

It should be noted that the instrument operating conditions noted above are preliminary. The conditions will be modified in the field, as necessary, to optimize instrument performance.

* * * * *

SAMPLE COLLECTION & ANALYSIS

ANALYTES OF INTEREST

Soil gas, soil and groundwater samples will be collected from the sites of interest. The target compounds analyzed for during this project are cis-1,2-dichloroethene (cis-1,2-DCE), trichloroethene (TCE), tetrachloroethene (PCE), and carbon tetrachloride (CCL4). For optimum detection of the analytes of interest, the gas chromatograph (GC) will be equipped with a photoionization detector (PID) and an electron capture detector (ECD) in series. The ECD is extremely sensitive to TCE and PCE and will be used as a confirmation back-up tool, while the PID will be calibrated to and used for actual analysis of cis-1,2-DCE, TCE, and PCE. The ECD will be used to quantify CCL4.

SAMPLE COLLECTION PROCEDURES

To minimize cross-contamination of samples, each test hole is probed with pre-cleaned sampling rods. Pre-cleaning consists of steam-cleaning, washing with Alconox soap, and rinsing with deionized water. Enough probe rods are available so that they are used only once between decon periods. In addition, disposable gloves, polyethylene tubing, and syringes are used for sample collection and analysis to minimize cross-contamination. All disposable equipment is used only once.

Procedures specific to the collection of soil gas, soil and groundwater are detailed in the following paragraphs.

Soil Gas Sampling Procedure

Soil gas sampling procedures include advancing 1.25-inch outer diameter (O.D.) probe rods with a threaded point holder and disposable point to the desired sampling depth on each test hole. Polyethylene tubing (1/4" dia.) is then lowered down the inner diameter of the probe rods and threaded onto the threaded point holder. An o-ring is utilized to ensure a tight seal surrounding this threaded fitting. The probe unit is then used to retract the rods approximately 2- to 4-inches, thereby disengaging the disposable drive point attached to the threaded point holder. Once a space or void has been made for soil gas extraction, the tubing is purged, utilizing the on-board vacuum system, to remove any extraneous air present in the polyethylene tubing.

Once purged, a soil gas sample is collected using a disposable syringe. The soil gas sample is collected by first pinching off the upper sample tubing, puncturing the tubing directly above the upper probe rod with the syringe, and evacuating 0.20 cc of soil gas from within the tubing into the syringe. The sample, 0.20 cc of soil gas, is then immediately injected into the GC for analysis.

Soil Sampling Procedure

Soil sampling procedures include vertically profiling the soils to the desired sampling depths or probe refusal. Continuous profiling procedures include attaching a 4-ft. in length Geoprobe™ macro-core soil sampler to the leading probe rod, and advancing the sampler in 4-ft. increments from the ground surface to the desired sampling depths. Discreet profiling procedures include attaching a closed piston-assembled 4-ft. in length Geoprobe™ macro-core soil sampler to the leading probe rod, and advancing the sampler in 4-ft. increments from the ground surface to the desired sampling depth. Once at depth, small diameter

(1/8") extension rods are lowered down the I.D. of the probe rods until they make contact with the piston rod and the reverse-threaded, piston rod stop pin. The stop pin is unthreaded freeing up the inner piston rod and driving point. The 4-ft. sampler is then advanced filling the sample tube with the discreet sample. The sampler is equipped with an inner PVC, transparent liner, useful for logging purposes. The filled liners are placed on visqueen and placed on the ground surface for visual observation. Soil samples are then collected and logged by Client personnel. Once a selected sample has been determined, EPS will conduct the on-site analysis of the soil samples. Selected duplicate samples will be sent from the Client to a certified laboratory for analysis.

Groundwater Sampling Procedure

Once it has been determined that depth to groundwater is within sampling range of the hydraulic probe and that sufficient water is available for sampling, groundwater samples will be collected for analysis. A 1-inch by 2-foot length of mill-slotted groundwater sampling tool, which is attached to the leading probe rod, is advanced in 3-foot sections to the desired sampling depth at each test hole. A larger diameter (1.5-inch) pre-probe point is connected to the bottom end of the mill-slotted tool. This pre-probe point is used to prevent the mill-slots from plugging with sediment as the rods are advanced to depth. If conditions do not warrant mill-slot usage due to the presence of too much clay or too small fines, then a Geoprobe™ SP-15 Screen Point Sampler will be used. This sampler is equipped with an inner 4-foot length of mesh-wire screen. Once the rods and SP-15 sampler are advanced to depth, the rods are retracted approximately 4 feet, which exposes the inner screen. Once groundwater has infiltrated the sampler, a groundwater sample can be collected.

Prior to sample collection, a groundwater depth measurement will be taken using a Solinst water level indicator. Static water levels are taken by lowering the water level indicator inside the probe rods until contact with water occurs. The water level is then measured to within 1/100th of an inch.

To collect the groundwater sample, the on board vacuum/volume system, a peristaltic pump, or a ball-and-seat check valve system is used. Whichever system is utilized, depending on hydrologic conditions, 3/8" polyethylene tubing will be used to collect the groundwater samples. The 3/8" tubing is lowered down the inside of the probe rod until it reaches the bottom of the mill-slotted section or SP-15 sampler. An up-down motion of the tubing allows groundwater to infiltrate up through the tubing to the ground surface if the ball-and-seat check valve system is used. Utilizing the vacuum/volume system, groundwater is extracted through the tubing by using a vacuum of no more than 21 inches of mercury. Once a sufficient amount of the groundwater has been purged from the sampling string, the tubing is pinched off at the ground surface and pulled from the probe rods. Groundwater is then immediately transferred from the lower end of the tubing directly into 40-ml vials. The vials are filled to minimize the headspace, and the samples are placed on ice and set aside awaiting analysis. Depending upon groundwater availability, a minimum of two 40-mL vials is typically collected at each sample location.

VERIFICATION OF REPORTING LIMITS

The reporting limit for each compound is determined by the sensitivity of each detector that is utilized. For example, based upon instrument performance using heated headspace the PID is typically sensitive to cis-1,2 DCE, TCE and PCE at or below 2 parts per billion (ppb). Similarly, the ECD is sensitive to CCL4 at or below 2 parts per billion. Therefore, the reporting limits for cis-1,2 DCE, TCE and PCE would be anticipated to be at 2 ppb in non-diluted samples. When running a different detector, or dual detectors, the same idea would apply to the ECD or FID pertaining to compounds to be screened. Prior to field activities, the reporting limits will be verified as described in the following paragraphs.

Verification of Soil Gas Reporting Limits

To verify the reporting limit, a standard is made for each analyte of interest, or a commercially prepared standard is utilized. The gas standard is made from stock solutions of the target compounds preserved in methanol. The distributor (Chem Service) guarantees the concentrations of the stock solutions (including DCE, TCE, PCE and CCL4) are 100ug/ml plus or minus 5% in methanol. To produce a 2 ppb gas standard, 20 microliters (µl) of each stock solution is injected into a 1000-ml glass bulb. To produce a 5 ppb gas standard, 50 µl of the stock solution is injected into a 1000-ml glass bulb, and so on.

The verification of the reporting limit is made by analyzing three (3) injections of the standard prepared at or below the chosen reporting limit (i.e., 2 ppb). If the instruments signal to noise ratio is 2.5 or greater, then the reporting limit has been verified. If this criterion is not met, then the reporting limit should be increased accordingly.

In order to meet data input requirements for the Shimadzu and P/E Nelson Analytical Data Processors, the average retention time and peak area is calculated for each analyte of interest during the verification process. The result of an internal 3-point calibration curve automatically determines a response factor, which enters into the Identification Table of the Data Processor. The response factor, as defined/required by the Data Processor, is determined by dividing the known concentration of each individual compound within the standard by its average peak area. Appropriate retention time windows are selected for the identification of each compound, based on degree of separation.

Verification of Groundwater/Soil Reporting Limits

To verify a 2 µg/L reporting limit, a standard is made for each analyte of interest. The liquid standard is made from stock solutions of the target compounds preserved in methanol. The stock solutions contain concentrations of the stock solutions (including DCE, TCE, PCE, and CCL4) at 100ug/ml plus or minus 5% in methanol**.**

A 1:5 Working Standard is prepared using the stock solutions. Standards are prepared in a 50cc volumetric flask for each calibration level using this compound mixture. Three 40-ml vials are then prepared by transferring 20-ml of the Working Standard from the volumetric glassware. Standards may also be prepared by spiking a half full 40-ml vial with the compound mixture. The vials are placed in a temperature-controlled oven at approximately 90°C for approximately 15 minutes. Once a vial has been thoroughly heated, 0.20 cc of headspace from within the vial is collected with a syringe and immediately injected into the GC for analysis.

The verification of the reporting limit is made by analyzing three (3) injections of a standard prepared at or below the chosen reporting limit (i.e., 2 µg/L). If the instruments signal to noise ratio is 2.5 or greater, then the reporting limit has been verified. If this criterion is not met, then the reporting limit should be increased accordingly.

In order to meet data input requirements for the Shimadzu and P/E Nelson Analytical Data Processors, the average retention time and peak area is calculated for each analyte of interest during the verification process. The result of an internal 3-point calibration curve automatically determines a response factor, which enters into the Identification Table of the Data Processor. The response factor, as defined/required by the Data Processor, is determined by dividing the known concentration of each individual compound within the standard by its average peak area. Appropriate retention time windows are selected for the identification of each compound, based on degree of separation.

CALIBRATION OF GAS CHROMATOGRAPH

Prior to the start of field activities, the GC will be calibrated using a 3-point calibration. Concentrations of 2, 10, and 20 ppb are typically used for the initial calibration for fuel compounds and chlorinated solvents. Broader calibration ranges may need to be utilized if high concentrations of the analytes of interest are detected within the field. This may be accomplished by a calibration check of a concentration range equivalent to the high concentration sample. It is anticipated that the initial calibration may be sufficient for the entirety of this field activity. Calibrations will be re-established if necessary. The stability of the calibration will be assessed during the field program through the use of calibration checks (see Page 12,13 **Quality Control / Quality Assurance**).

A chromatograph and area report that was generated by the chromatography software will be provided for each standard that is analyzed. Calibration reports that are generated by the chromatography software will also be provided.

Calibration of Gas Chromatograph for Soil Gas Samples

Standards are commonly made at concentrations of 2, 10, and 20 ppb for each chlorinated analyte of interest. The gas standard is made from stock solutions of the target compounds preserved in methanol. Concentrations of the stock solutions (including DCE, TCE, PCE and CCL4) are 100ug/ml plus or minus 5% in methanol. To produce each concentration of the gas standard, the appropriate volume of each stock solution is injected into a 1000-ml glass bulb.

Once the gas standard has been made, a known volume (typically 0.20 cc) of the gas standard is injected into the GC for each concentration. A calibration factor is calculated for each concentration of each target compound. The ratio of the detector response (peak area or peak height) to the concentration of analyte in the calibration standard is defined as the calibration factor. The CF is calculated as follows:

$$CF = \frac{\text{Peak Area (or Peak Height) of the Compound in the Standard}}{\text{Concentration of the Compound Injected (ppb or } \mu\text{g/L)}}$$

To evaluate the linearity of the initial calibration, the P/E Nelson integration software calculates a correlation coefficient (CC) by linear regression, from three calibration injections using a straight-line fit. The mean CF, the standard deviation (SD), and the relative standard deviation (RSD) can be manually calculated as follows:

$$meanCF = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n} = \frac{CF_{1ppb} + CF_{5ppb} + CF_{10ppb}}{3}$$

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}\right)^2}{n-1}} = \sqrt{\frac{\left(CF_{1ppb} - \overline{CF}\right)^2 + \left(CF_{5ppb} - \overline{CF}\right)^2 + \left(CF_{10ppb} - \overline{CF}\right)^2}{2}}$$

$$RSD = \frac{SD}{\overline{CF}} \times 100$$

Note: In the preceding calculations *n* is the number of calibration standards and RSD is expressed as a percentage (%).

If the RSD of the calibration factors is less than or equal to 25% over the calibration range for each compound of interest, then linearity through the origin is assumed. The average calibration factor (mean CF) can then be used to determine sample concentrations by manual calculation.

If the RSD for any target analyte exceeds 25%, then one or more of the following corrective actions will be necessary and will be performed in the following order:

- Check the instrument operating conditions
- Review the results (peak area) and subsequent calculations that do not meet the linearity criteria. If the problem appears to be associated with a single standard or injection, that standard/injection may be reanalyzed and the calibration statistics recalculated.
- Re-calibrate until all compounds meet the linearity criteria.

Calibration of Gas Chromatograph for Groundwater/Soil Samples

Standards are commonly made at concentrations of 2, 10, and 20 µg/L for each analyte of interest. Please note that calibration ranges may be increased within the field if high concentrated samples detected cannot be diluted within the 20 ppb calibration range. This may be accomplished by a calibration check of a concentration range similar to the high concentration sample detected within the field. The liquid standards are made from stock solutions of the target compounds preserved in methanol. The stock solutions contain concentrations at 100ug/ml plus or minus 5% in methanol. A 1:5 Working Standard stock mixture is prepared using these stocks.

Working Standards for each calibration concentration (i.e. 2, 10, and 20 µg/L) are prepared from Intermediate Standards. Following preparation in a 50 cc volumetric flask, 20-ml of each calibration concentration is transferred to a 40-ml vial. The standard can also be prepared by spiking a half full 40-ml vial with the compound mixture. The vials are placed in a temperature-controlled oven at approximately 90°C for approximately 15 minutes. Once a vial has been thoroughly heated, 0.20 cc of headspace from within the vial is collected with a syringe and immediately injected into the GC for analysis.

Linear regression is then used by the Nelson software to evaluate the linearity of the initial 3-point calibration by internal calculation of a correlation coefficient (r) that represents the "fit" of a straight line to the data. A value of r=1.00 indicates a perfect fit. Calibration is determined acceptable when $r \geq 0.98$. Alternatively, the coefficient of determination (r^2) can be determined. Calibration is determined acceptable when $r^2 \geq 0.97$. Calibration of certain compounds with low detector response or short shelf life may be considered acceptable at a slightly lower coefficient. The Client will approve any deviance from the $r \geq 0.98$ (or $r^2 \geq 0.97$) criteria prior to analysis of any samples.

While the linear regression is performed internally by the Nelson software, it can also be performed manually using statistical procedures. The linear regression calculations produce the slope and y-intercept of a line of peak area versus compound concentration. The linear equation is as follows:

$$y = ax + b$$

Where:

y = Peak area or height
a = Slope of the line
x = Concentration of target compound
b = y-intercept

Alternatively, linearity of calibration can be manually evaluated using the average CF (CF). Equations for the manual calculation of the mean CF, the standard deviation (SD), and the relative standard deviation (RSD) are used.

Once each standard has been analyzed, a calibration factor is calculated (Shimadzu software) for each concentration of each target compound. The equations for the mean CF, the standard deviation (SD), and the relative standard deviation (RSD) can be done manually as previously presented (See Page 8, **Calibration of Gas Chromatograph for Soil Gas Samples**).

FIELD SCREENING & ANALYSIS

Soil Gas Analysis

At the beginning of each day of analysis, a gaseous calibration check and method blank analysis will be performed. (See Page 12, 13) **Quality Control/Quality Assurance**, for further discussion of calibration checks and method blanks.) Once it has been established that both the calibration check and method blank meets the acceptance criteria, field samples are analyzed. As previously presented, a soil gas sample is collected from the sampling tubing using a disposable syringe. The sample, 0.20 cc of soil gas, is then immediately injected into the GC for analysis. After the analysis of 10 field samples, the method blank analysis is repeated. The calibration check is repeated at the end of each day.

Once the calibration is established, or the response factor defined (Shimadzu software) and retention time for each individual compound has been entered into the ID table of the GC, the GC's Data Processor identifies each compound and reports the concentrations directly. The concentration of an analyte in the sample can also be calculated manually using the following equation:

$$Concentration, ppb = \frac{(A_s)(D)}{(\overline{CF})}$$

Where:

A_s = Peak area for the analyte in the sample

D = Dilution factor (dimensionless). If no dilution was necessary, D=1.

CF = Mean calibration factor from the initial gas calibration (area per ppb)

If a sample exhibits a concentration that potentially saturates the detector, dilution will be necessary. For soil gas samples, dilution refers to injection of a smaller volume of soil gas into the GC.

Soil Analysis

At the beginning of each day of analysis, an aqueous calibration check and method blank analysis will be performed. (See Page 12, 13) **Quality Control/Quality Assurance**, for further discussion of calibration

checks and method blanks.) Once it has been established that both the calibration check and method blank meets the acceptance criteria, field samples are analyzed. Following soil collection, 5 grams of soil are transferred into a separate 40-ml vial. The vial is then filled to the half-full mark with deionized water (approx. 20 mLs.). The vial is then shaken vigorously dissolving the soil into the DI water. The vial is then placed into a temperature-controlled oven at 90°C for approximately 15 minutes. Once heated, the vial is removed from the oven, then 0.20 cc of headspace from within the vial is collected with a disposable syringe, and the sample is immediately injected into the GC for analysis. After the analysis of 10 field samples, the method blank analysis is repeated. The calibration check is repeated at the end of each day.

Once the calibration and/or response factor (defined previously) and retention time for each individual compound has been entered into the ID table of the GC, the GC's Data Integrator identifies each compound and reports the concentrations in µg/L. The concentration of the compounds within the soil is then converted to µg/Kg by multiplying the concentration reported in µg/L by the total volume of headspace (21 mL, actual) within the vial divided by the weight of the soil collected and analyzed (5 grams).

If a sample exhibits a high concentration that will potentially saturate the detector, dilution may be necessary. Using the "back-up" vial collected at the sampling location, dilution will be performed by transferring less than 5 grams of soil into a 40-mL vial and adding deionized water until the total volume reaches the half-full mark on the vial. The diluted sample is then analyzed as previously described. As an alternative to preparing a dilution, a smaller volume of headspace could be injected into the GC. In some cases, less soil may be used to increase dilution ranges.

Groundwater Analysis

At the beginning of each day of analysis, an aqueous calibration check and method blank analysis will be performed. (See Page 12, 13) **Quality Control/Quality Assurance**, for further discussion of calibration checks and method blanks.) Once it has been established that both the calibration check and method blank meets the acceptance criteria, field samples are analyzed. Following groundwater collection, 20 ml of groundwater is transferred into a separate 40-ml vial. The vial is then placed into a temperature-controlled oven at 90°C for approximately 15 minutes. Once heated, the vial is removed from the oven, 0.20 cc of headspace from within the vial is collected with a disposable syringe, and the sample is immediately injected into the GC for analysis. After the analysis of 10 field samples, the method blank analysis is repeated. The calibration check is repeated at the end of each day.

Once the calibration and/or response factor (defined previously) and retention time for each individual compound has been entered into the ID table of the GC, the GC's Data Integrator identifies each compound and reports the concentrations directly. The concentration of an analyte in the sample can also be calculated manually using the following equation:

$$Concentration, ug/L = \frac{(A_s)(D)}{(\overline{CF})}$$

Where:

A_s = Peak area for the analyte in the sample

D = Dilution factor (dimensionless). If no dilution was necessary, D=1.

CF = Mean calibration factor from the initial aqueous calibration (area per µg/L)

If a sample exhibits a concentration that may potentially saturate the detector, dilution will be necessary. Using the "back-up" vial collected at the sampling location, dilution will be performed by transferring less than 20 ml of groundwater into a 40-mL vial and adding deionized water until a total volume of 20 ml of liquid is in the vial. For example, a dilution factor of 2 would require 10 ml of groundwater sample and 10 ml of deionized water. Likewise, a dilution factor of 5 would require 4 ml of groundwater sample and 16 ml of deionized water. The diluted sample is then analyzed as previously described. As an alternative to preparing a dilution, a smaller volume of headspace could be injected into the GC, or these procedures may be used in combination.

QUALITY CONTROL / QUALITY ASSURANCE

Calibration Check

Calibration checks are performed to ensure that the relationship between instrument response and initial calibration is valid over the duration of the field effort. Calibration check samples will be analyzed at the beginning and end of each day of analysis. For the gaseous and aqueous calibration checks, a mid-point standard will be utilized. Aqueous standards are prepared on-site. The calibration checks regarding both aqueous and gas standards for each target analyte must fall within 25% of the true value. If this criterion is not met, then a second calibration check standard will be analyzed. If the second standard also falls outside of acceptance criteria, then the standards may be used to re-calibrate prior to further sample analysis. If field screened samples exhibit analyte concentrations greatly exceeding initial calibration ranges, calibration checks may include higher concentrations that reflect these levels.

Blank Samples

Method Blanks

Method blanks will be run at the beginning of each day and following every 10th sample to ensure a clean system. For soil gas samples, the method blank consists of ambient air collected in a disposable syringe. For groundwater samples, the method blank consists of reagent water placed in a 40-mL vial and heated in the temperature-controlled oven for approximately 15 minutes at 90°C. Once heated, 0.20cc of headspace from the vial is injected into the GC. If a detection is noted within a method blank, then the blank analysis will be repeated. If the associated field samples have similar detections, these samples will be reanalyzed if sufficient back-up volume was able to be collected.

Rinsate Blanks

Prior to the commencement of, or during sampling, rinsate blanks will be collected from syringes, soil/groundwater sampling tools, stirring rods, and/or sample vials to ensure that no cross-contamination has occurred. If compound detection is noted within a rinsate blank, then the equipment will be re-cleaned. If the associated field samples have similar detections, these samples will be reanalyzed if sufficient back-up volume was able to be collected.

Duplicate Samples

Duplicate samples are collected and analyzed to demonstrate the precision of the collection and analysis process. Duplicate samples are collected and analyzed at a frequency of 10% of the total samples

collected. Since duplicate sample results are dependent on both collection and analysis, the optimal deviation for duplicate samples is a relative percent difference (RPD) of less than or equal to 25%. The RPD is calculated as follows:

$$RPD = \frac{|C_1 - C_2|}{\frac{(C_1 + C_2)}{2}} \times 100$$

Where:

C_1 = Measured Concentration of the first sample
C_2 = Measured Concentration of the duplicate sample
RPD = Relative Percent Difference

If the RPD between duplicate samples dramatically exceeds 25% and sufficient "back-up" sample volume was able to be collected, the sample may be reanalyzed.

* * * * *

APPENDIX C

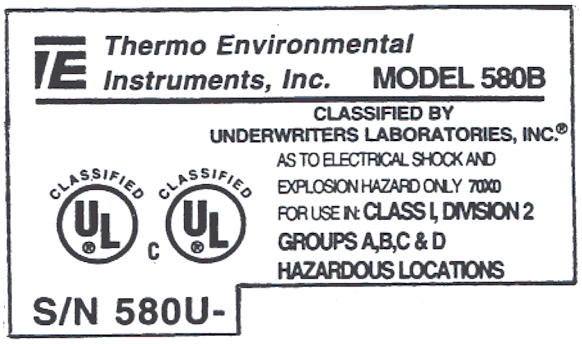
FIELD EQUIPMENT MANUALS

Model 580B
Organic Vapor Meter (OVM) / Datalogger
Instruction Manual

MODEL 580B

ORGANIC VAPOR METER (OVM) / DATALOGGER

INSTRUCTION MANUAL
P/N 16860



THIS EQUIPMENT IS SUITABLE FOR USE IN CLASS I, DIVISION 2, GROUPS (AS APPLICABLE) OR NON-HAZARDOUS LOCATIONS ONLY.

WARNING - EXPLOSION HAZARD - SUBSTITUTION OF COMPONENTS MAY IMPAIR SUITABILITY FOR CLASS I, DIVISION 2.

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FACSIMILE: (508) 520-1460

1-9-96

INSTRUMENT WARRANTY

WARRANTY. Subject to the exceptions stated below, Thermo Environmental Instruments Inc. agrees to correct, either by repair or at our opinion, by replacement, any defects in materials or workmanship which develop within one year from the date of surface shipment, parts and labor supplied free of charge and surface transportation costs to be borne by the offeror both ways, provided that the investigation and inspection defects developed under normal and proper use.

The exceptions mentioned above are: (1) All items claimed must be returned to Thermo Environmental Instruments Inc., transportation charges collect, and will be shipped prepaid and charged to the customer unless the item is found to be defective and covered by the warranty in which case Thermo Environmental Instruments Inc. will pay all surface transportation charges; (2) Thermo Environmental Instruments Inc. agrees to extend to the customer whatever warranty is given to Thermo Environmental Instruments Inc. and incorporated into products sold to the customer; (3) Thermo Environmental Instruments Inc. shall be released from all obligations under this warranty in the event repairs or modifications are made by persons other than its own authorized service personnel, or service personnel from an authorized representative, unless such repair is minor, merely the installation of a new plug-in component; (4) If any model or sample was shown to the Purchaser, such model or sample was shown merely to illustrate the article and not to represent that any article delivered hereunder would conform to the model or sample; and (5) Spare parts are warranted for ninety (90) days.

THE FOREGOING WARRANTY IS EXCLUSIVE AND IN LIEU OF ALL OTHER WARRANTIES, WHETHER WRITTEN, ORAL, IMPLIED OR STATUTORY. SELLER DOES NOT WARRANT MERCHANTABILITY OR FITNESS FOR ANY PARTICULAR PURPOSE, OR MAKE ANY OTHER WARRANTY OR AGREEMENT EXPRESSED OR IMPLIED WITH RESPECT TO ANY ARTICLES COVERED HEREUNDER. tHERE ARE NO WARRANTIES WHICH EXTEND BEYOND THOSE EXPRESSLY STATED IN THIS CONTRACT.

580B USER MANUAL
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SECTION I

INTRODUCTION

1 INTRODUCTION

The 580B is a portable Organic Vapor Meter (OVM), which detects and quantitates most organic vapors with a highly sensitive photoionization detector (PID). The 580B has an operating range of 0-2000 parts per million (ppm) with a minimum detectable of 0.1 ppm. No support gases are required.

The 580B is controlled by a microprocessor which provides many features that were not previously available. Maximum signal hold, detector linearization, overrange lockout, IBM PC (or compatible) interface, extensive data logging capabilities and much more. With the many features provided by the 580B leak detection, head space measurements, and field survey are all easily accomplished. Completely portable, the 580B operates from internal batteries for eight hours in the field.

1.1 ABOUT THIS MANUAL

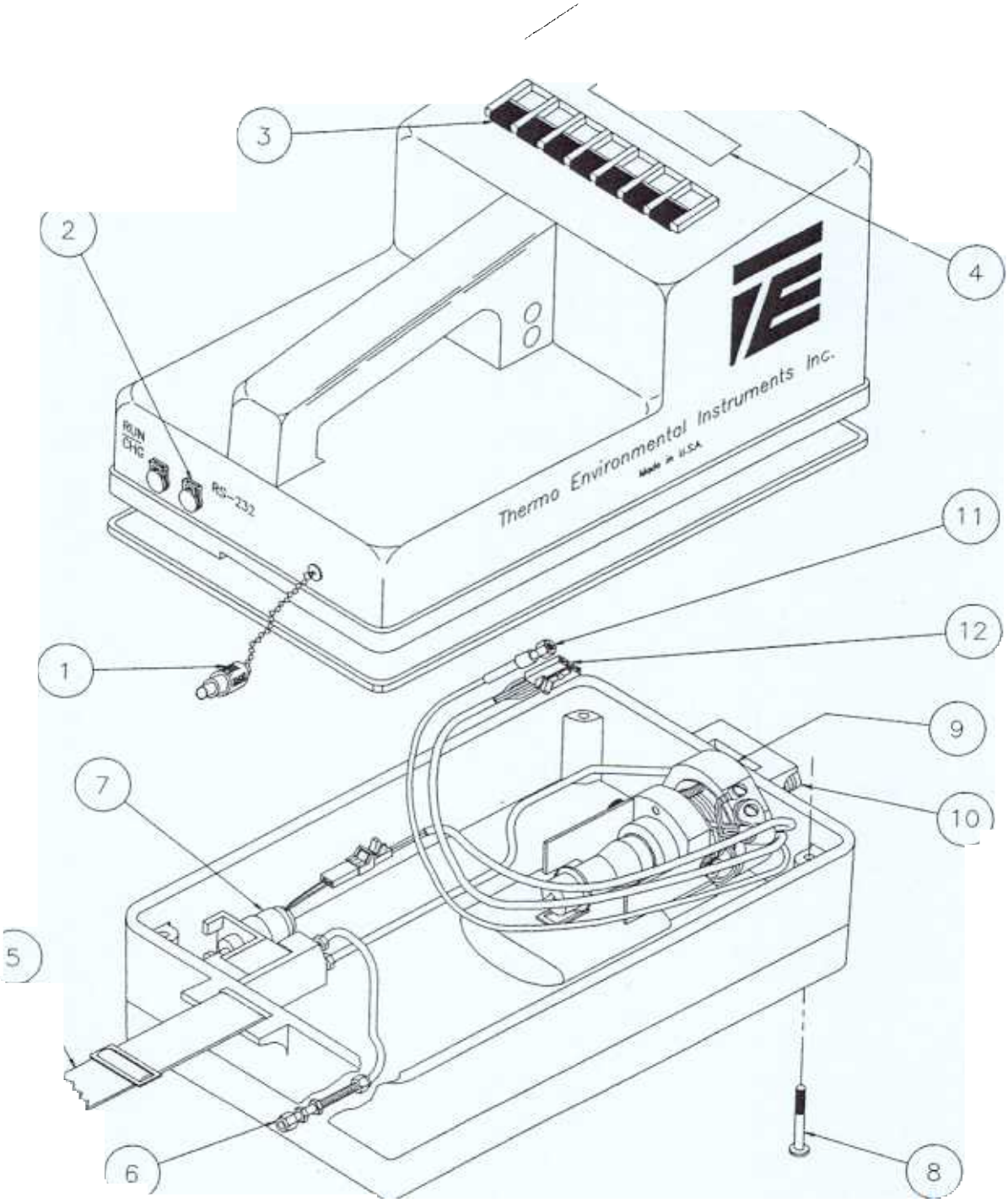
This manual is broken down into eight chapters. The first chapter (this one) provides a general overview of the 580B. Chapter two discusses, in great detail, the extensive facilities of the 580B. The focus of this chapter is on how to use the seven switches to access the various facilities. Chapter three explains, in detail, how to perform routine maintenance on the 580B. Chapter four is a technical discussion of calibration and methods for generating standards. Chapter five is a technical discussion of a few applications which illustrate some of the uses of the 580B. Chapter six is a technical discussion of methods for collecting a sample using the 580B. Chapter seven is a discussion of the communication facilities provided by the 580B. Chapter eight contains two flow charts which illustrate the 580B software flow. This chapter is a helpful tool for the new user. Appendix A is a detailed explanation of the 580B communication protocol. This chapter is provided in order to allow a programmer to develop specialized communication software for the 580B. There are several other addendums which contain miscellaneous information about the 580B.

1.2 INSTRUMENT OVERVIEW

This section describes various points of interest on the 580B. Each number refers to a number in Figure 1.1.

1. **POWER PLUG** - The power plug is used to run the instrument from its internal batteries. There is a chain attached to the power plug so that it will not be lost.

2. **RS-232 CONNECTOR** - This connector is used for communi-



Figur
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cation with a serial printer or computer. A communication cable provided with the instrument fits into the receptacle.

3. **KEY PAD** - There are seven switches which operate the 580B. The switch marked ON/OFF is used to turn the pump and lamp on and off. The switch marked LIGHT will turn on backlighting for the two line display. The other five switches perform various functions. For a detailed description of the function of each switch see chapter two or the flow charts in chapter eight.

4. **DISPLAY** - The 580B has a two line by sixteen character display.

5. **SHOULDER STRAP** - There is an adjustable shoulder strap for carrying the 580B.

6. **SAMPLE EXIT PORT** - The 580B sample is drawn into the detector by a positive displacement pump and then sent back out through the exit port.

NOTE : The photoionization detector is a non destructive detector so the sample may be collected at the exit for further analysis (see Chapter Six).

7. **PUMP** - The 580B pump draws the sample into the detector.

8. **MOUNTING SCREWS** - There are four captive screws which hold the 580B top and bottom together. The screws are specially designed so that they do not fall out when they are loosened out of the case top.

9. **DETECTOR** - The photoionization detector is shown with the lamp and high voltage power supply.

10. **SAMPLE INLET** - Sample is drawn into the detector through the sample inlet at the front of the 580B.

11. **SIGNAL CABLE** - The PID signal is brought up to the microprocessor, for analysis, via the coaxial signal cable.

12. **BASE HARNESS** - The base harness plugs into a connector on the case top.

1.3 580B FEATURES

This section provides a brief overview of the various features of the 580B. After reading this section the user should have a good idea of what the instrument can do. Chapters two and three will explain, in detail, how each feature is selected.

TURNING ON PUMP AND LAMP - The pump and lamp are turned on by pressing the ON/OFF switch (the instrument power must already be on).

CALIBRATION - Calibration of the 580B is extremely impor-

tant. Chapter two explains how to calibrate the 580B in great detail. Chapter four discusses at length some of the basic theory and methods behind calibration. It is strongly suggested that this chapter be read in order to gain a deeper understanding of usage of the 580B. Chapter three also discusses calibration.

CONCENTRATIONS - Once the lamp and pump have been turned on the 580B begins to display the concentration of the incoming sample on the bottom line of the display. Normally the top line of the display will be a bar graph (logarithmic on a scale of zero to 2000). The operator may however select the MAX HOLD mode of operation. When in MAX HOLD, the top line of the display will show the highest concentration recorded.

LOGGING - The 580B provides extensive facilities for logging information. The operator may save a particular reading along with a six digit location code and a date and time stamp. If the 580B is in the MAX HOLD mode when logging is initiated then the max hold value will be logged.

AUTO LOGGING - Logging may be performed automatically by using the 580B's auto logging feature. Auto logging is not allowed while in the MAX HOLD mode. When auto logging is selected a LOGGING INTERVAL is selected (anywhere from one second to 99 minutes and 59 seconds). At the end of each logging interval the present concentration will be logged (the location code is automatically incremented each time).

AVERAGE - The 580B normally updates the concentration once per second. The operator has the option of setting the averaging time anywhere from one second up to four minutes.

NOTE : The bottom line of the display will be blank until the first averaging interval is completed. The top line will however be updated each second.

RESPONSE FACTOR - A response factor may be used in order to relate a particular gas to the calibration gas. When computing the displayed concentration the microprocessor multiplies the measured concentration by the response factor and displays the result. If the response factor is one, then the concentration is not changed. Chapters four and five explain some uses of the response factor.

LAMP SELECTION - The 580B allows for calibration data to be saved for one 10.0 eV lamp and one 11.8 eV lamp. This allows lamps to be switched in the field without requiring recalibration. A lamp serial number may also be entered.

ALARM - An alarm level may be selected. The 580B will sound an audible alarm (the top line will also indicate an alarm) whenever the concentration goes above the selected alarm level.

ACCESS - The 580B provides four access levels so that various features may be "locked out." User identification number

and instrument number are also provided.

CLOCK - The 580B has an internal clock which will run even when the instrument power is cut off.

COMMUNICATION - The 580B has a serial communication port for outputing data to a serial printer. Many of the 580B features may be accessed from a remote computer through the serial communication port (there is communication software available which will run on an IBM PC or clone).

DISPLAY LOGGED DATA - The logged data may be displayed on the 580B's two line display.

SECTION II

PRINCIPAL OF OPERATION

PHOTOIONIZATION DETECTOR OPERATION & THEORY

1 GENERAL

The sample is drawn into the ion chamber by a pump down stream of the detector. Here the sample is bombarded by ultra-violet light (UV) exciting the molecule. If the energy, (hU) of the UV light is greater than the ionization potential (IP) of the sample molecule (R) an electron will be removed, ionizing the molecule. A positively charged molecule and a free electron are produced, as :

$$R + hu \rightarrow R^{+} + e^{-}$$

Several typical reactions follow:

C_6H_6 (benzene) + hu -> $C_6H_6^{+}$ + e^{-}	IP = 9.2 eV
$H_2C{=}CHCl$ (VCM) + hu -> $H_2C{=}CHCl^{+}$ + e^{-}	IP = 9.9 eV
C_3H_8 (propane) + hu -> $C_3H_8^{+}$ + e^{-}	IP = 10.9 eV

For this reason the ionization potential of the subject molecule plays an important role in selecting the lamp energy. Ionization potentials are expressed in electron volts (eV). A list of ionization potentials can be found in Appendix E of this manual or a more complete list in the CRC "Handbook of Chemistry and Physics".

2.1.2 LAMP ENERGIES

There are three lamps available from TEI, 10.0 eV, 10.6 eV, and 11.8 eV. The different energies are obtained by filling the lamp envelopes with different gases and selecting a window which will pass the wavelength produced when the gas is excited. The combination of gas and windows which produce these energies are listed below:

ENERGY	GAS	WINDOW	WAVELENGTH (nm)
10.0 eV	Krypton	MgF	123.6
10.6 eV	Krypton	MgF	117.4
11.8 eV	Argon	LiF	105.1

Though ionization potential will help the user select a lamp, it will not give any information as to the performance of

the detector in measuring a specific compound. The response of the system varies considerably from compound to compound even though they may have the same ionization potential. Some generalizations may help the user obtain a feeling for the difference in response between compounds.

2.1.3 COMPARATIVE RESPONSE

The following is an idealized response chart. No attempt is made to quantitate the relationship, it's a guideline.

Decreasing PID Response:
- Aromatic Compounds
- Unsaturated Compounds
- Saturated Compounds
- Ketones
- Alcohols
- Compounds with Sub Groups

It becomes obvious that sensitivity is influenced by the electronegativity of the molecule though this is not a predictable measure of performance. The only true test of performance is to measure the specific compound of interest and compare it to a good performing standard such as isobutylene.

2.1.4 RESPONSE FACTORS

This relative comparison with isobutylene mentioned above is a very effective way of measuring a variety of compounds without the need to recalibrate for each compound.

The development of a RESPONSE FACTOR allows the operator to correct the instrument's response given a one to one correspondence for all compounds measured, using isobutylene as the reference standard.

Note: Because there is variation in lamp production and hence performance, it is suggested that all calibration and subsequent development of response factors be done on the same lamp/instrument combination.

The preparation of standards and the development of response factors is discussed in subsequent sections of this manual. Once the response factor is generated, it is entered into the 580. The instrument automatically reports the concentration of the compound measured in relative units. It is important to recognize that all compounds measured at that time will be reported relative to the response factor entered in the instrument. For example, if we have calibrated the instrument on isobutylene and have entered a response factor for benzene, we will read concentrations with a one to one correspondence to benzene. If during these measurements toluene or any other compound is encountered, the instrument will report the concentration as if it was measuring benzene. For this reason care should be taken when using this facility.

The above discussion should give the reader a good overview of PID performance. To further understand the intricacies of the

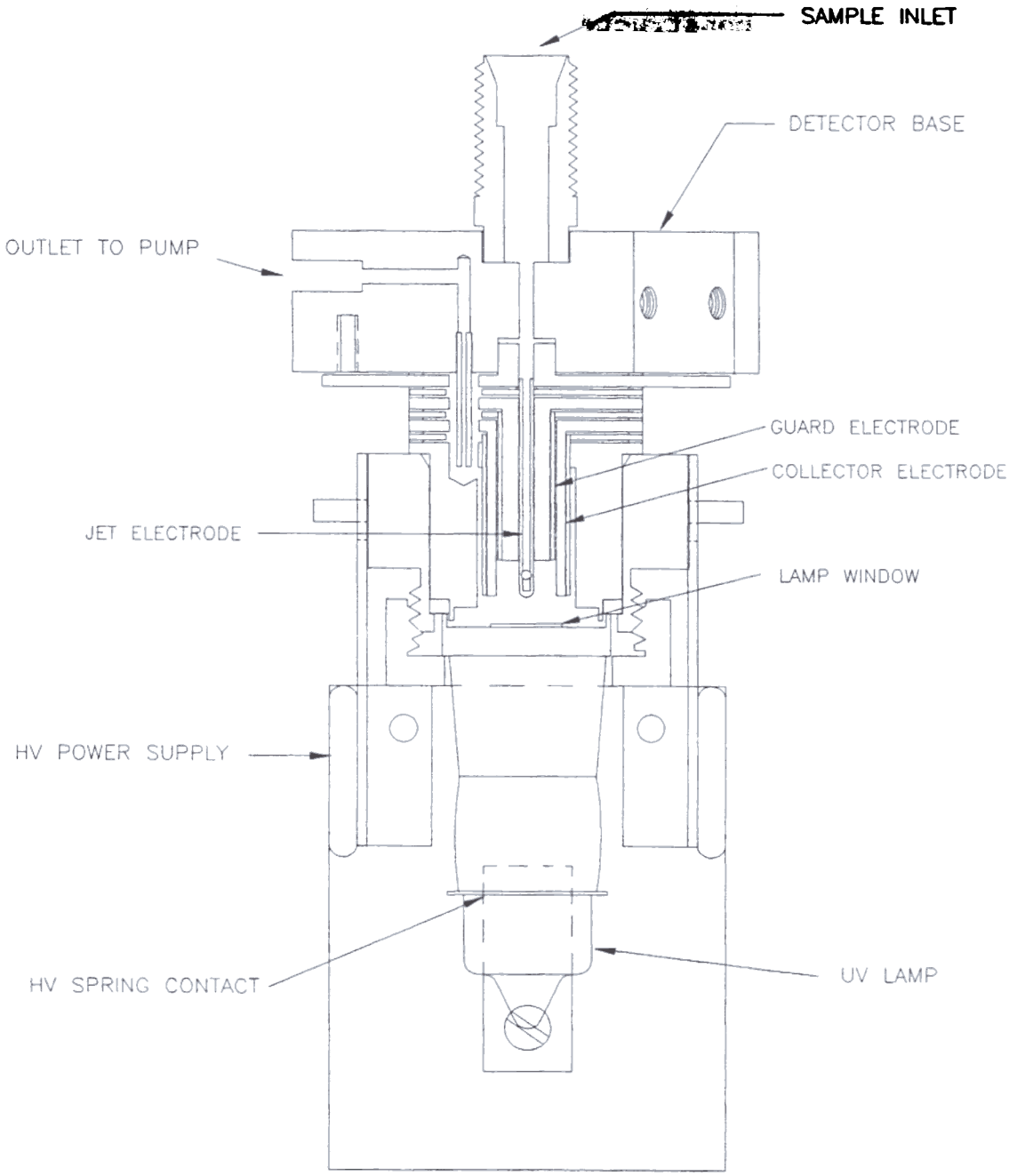


Figure 2.1
Photo Ionization Detector

instrument it is suggested that the user prepare a number of standards of different compounds and measure them relative to isobutylene. Included in this comparison should be several mixtures of compounds such as gasoline, paint thinner, or cleaning solvent, etc. Through this type of study the inequity of the PID response will be better understood making the Model 580 a more effective tool. The use of the instrument is discussed in greater detail in subsequent sections.

2.1.5 PHOTOIONIZATION DETECTOR

The detector is constructed of Teflon and stainless steel to eliminate chemical interaction with the surfaces that are encountered by the sample. To further reduce possible interaction with the surfaces, the flow rate thru the detector is high, 400 - 500 cc/min developing a very dynamic transport of the sample.

Referring to Figure 2.1, the sample is drawn into the ionization chamber through the jet electrode where the UV radiation from the lamp ionizes the sample. A bias voltage of several hundred volts is applied to the jet to aid in the collection of ions. As a result of the ionization process and the impingement of the UV energy from the lamp on the sample, positively charged ions and free electrons are produced. The jet is negative relative to the collector where the electrons are collected.

Between the jet and the collector, separated on both sides by Teflon, is the guard electrode. Its function is to eliminate surface currents which could flow between the two active electrodes. When the Teflon surfaces become dirty during use, there can be the development of a conduction path on the Teflon, which increases in high humidity situations. The guard electrode eliminates this path. The collector electrode is connected to the electrometer which measures the ion current produced during the ionization process. The sample is moved through the detector by an external pump which is on the exit of the detector.

2.2 PROGRAM OPERATION

2.2.1 INTRODUCTION

The 580B has seven switches located just below the display. They are labeled:

ON/OFF MODE/STORE RESET LIGHT +/INC -/CRSR SPKR

The ON/OFF switch toggles the lamp and pump power between on and off. The MODE/STORE, RESET, +/INC, -/CRSR and SPKR switches all have various meanings (including none at all) depending upon the mode. The SPKR switch normally is used to toggle the instrument speaker between on and off. Pressing the MODE/STORE switch will cause the 580B to return to the Run mode, except when the 580B is already in this mode. In which case it will cause the 580B to enter the Log mode.

The LIGHT switch is used to illuminate the display.

The 580B has several modes. Some of the modes may have sub modes. The modes and sub modes are tabulated below.

- Run mode
  - Concentration meter normal
  - Max hold
- Log mode
- Parameter mode
  - Calibration mode
- Access mode
- Clock mode
- Communication mode

The following sections will describe each mode and how to get to them and through them. It is strongly suggested that this section be carefully read and that the 580B be used along with the manual in order to re-enforce the manual.

2.2.2 POWER FOR LAMP AND PUMP

When the 580B is first turned on (see section 1.3) the display will indicate that the lamp is not lit. Pressing the ON/OFF switch will tell the microprocessor to turn on the lamp and the pump. The microprocessor will send power to the lamp and pump and then "look" to see if the lamp is actually lit. If it did not light then the microprocessor will try again.

If after fourteen seconds the lamp still will not light, then the microprocessor will indicate a lamp out condition.

In the event that the microprocessor is unable to light the lamp, check the seating of the lamp (see section 3.1). If the problem persists, call service.

Once the lamp is lit, the display will show the PPM (parts per million) on the bottom line. The top line will either be a bar graph or the maximum reading (see section 2.2.3).

To turn the lamp and pump off simply press the ON/OFF switch.

2.2.3 RUN MODES

The 580B has two run modes, Max Hold and Concentration meter. The run mode is selected in the Parameters section (see Section 2.4). In the concentration meter mode the top line of the display will be a bar graph. The bar graph is a logarithmic bar graph over the range of 0 to 2000 PPM. The bar graph is intended as a rough visual indication of the current PPM. The bottom line will indicate the exact PPM.

In the Max Hold mode the top line of the display will indicate the maximum reading. The bottom line of the display will indicate the current PPM. Whenever a new maximum is seen, the top line will be updated. The Max Hold reading may be reset by pressing the RESET switch while in the run mode.

2.2.4 LOG MODE

The ability to "log" data is one of the 580B's greatest

features. Readings may be stored for later analysis. Each reading will have a date and time as well as a location code associated with it. Up to over 700 readings may be stored. Logged data may even be sent to a printer or computer via an RS-232 serial communication port (see section 2.7).

The Log mode is entered from the Run mode by pressing the MODE/STORE switch. When this switch is pressed from the Run mode the display will show:

LOG THIS VALUE?

on the top line and either PPM or MAX PPM on the bottom line depending upon which run mode the 580B is currently in. By pressing the +/INC switch the display will then show:

LOC. CODE 000001

on the top line (the actual location code may not be 000001). The location code may now be entered. By pressing the +/INC switch the number above the cursor may be incremented. By pressing the -/CRSR switch the cursor may be moved to the next digit. The 580B automatically increments the location code each time a data point is logged.

Once the desired location code has been entered, pressing the MODE/STORE switch will "log" the data point. This means that the reading displayed on the bottom line, along with the location code, the current date and the current time will be stored into the 580B's memory. The 580B will then return to the Run mode.

If for any reason logging is not desired, pressing the RESET switch rather than the MODE/STORE switch will cause the value not to be stored. The 580B will then go back to displaying:

LOG THIS VALUE?

Pressing the mode switch will now return the 580B to the Run mode.

It is possible, when attempting to log a data point, that rather than the display showing "LOC. CODE 000001" it will show "BAR CODE: ." Don't be alarmed. This has happened becouse the location mode selection is not properly set. Section 2.4.3 describes how to set this parameter. Pressing the mode/store switch will cancel the logging operation and return to the run mode. The location mode selection should be changed as described in section 2.4.3.

2.2.4A AUTO LOGGING MODE

The 580B may be instructed to automatically log data according to a predefined time interval. AUTO LOGGING is selected from within the Parameters section (see section 2.4). At the end of the logging interval (settable from 1 second up to 99 minutes and 59 seconds) the current average ppm value will be logged and the logging interval will be restarted.

NOTE: Auto logging is not allowed with the Max Hold mode.

2.2.5 SPEAKER

While the 580B is in the Run mode the speaker may be turned on. The speaker will generate a "clicking" which will increase in speed as the concentration increases. The purpose of the speaker is to give the operator an audible indication of the PPM. The speaker may be turned on or off by pressing the SPKR switch. The speaker rate may also be changed by changing the switches located inside of the instrument. Only one of the four speaker rate switches should be on (in the down position) at any time.

2.2.6 LOW BATTERY

The 580B will display a warning when the battery is low. The warning will be a flashing B in the left hand corner of the bottom line of the display when the 580B is in the Run mode. The 580B should be recharged when the low battery warning is activated.

2.2.7 OVERRANGE

The 580B will display an overrange warning if the concentration goes above 2000 PPM. The top line of the display will show:

OVERRANGE

Once an overrange condition occurs the instrument will "lock out". This means that the overrange warning will continue to be displayed until the instrument is brought to a "clean" area. A clean area is defined to be an area where the concentration of organic vapors is below 20 PPM. The 580B will continue to indicate PPM on the bottom line during an overrange condition.

2.2.8 ALARM

The 580B has an alarm which will sound if the PPM rises above the alarm setting. The alarm setting is entered in the Parameters mode (see section 2.4.3). If the speaker is not activated then the alarm will of course not be heard. Once the PPM drops below the alarm setting the alarm will turn off. The top line of the display will also indicate when there is an alarm condition.

MAIN MENU

By pressing the MODE/STORE switch from the Run mode and then pressing the -/CRSR switch when asked if logging is desired,

580B will display the main menu:

R/COMM	-/PARAM
+/ACCESS	S/CLOCK

The other four operating modes (Communication, Parameters, Access and Clock) may be entered from the Main menu. The operating mode may always be returned to by pressing the MODE/STORE switch.

2.4 PARAMETERS MODE

All of the 580B operating parameters are entered in the Parameters mode. The 580B is also calibrated from within the Parameters mode.

The Parameters mode may be entered by pressing the -/CRSR switch from the main menu.

There are nine different sections in the Parameters mode.

1. Run mode selection
2. Auto logging selection
3. Location mode selection
4. Average time selection
5. Alarm setting
6. Lamp selection
7. Response factor setting
8. Calibration
9. Free space indication

Pressing the +/INC switch will advance the 580B to the next section. Pressing the -/CRSR will advance the 580B to the previous section. Each section and any of its sub-sections will be described in the following pages. It is important to note that when the 580B is in a sub-section of any of the above sections that the +/INC and -/CRSR switches will have a different meaning. This may seem confusing at first but will become clear after stepping through each section.

2.4.1 RUN MODE SELECTION

There are two Run modes. Concentration meter normal and Max Hold (see Section 2.2.3). The top line of the display will show:

CONC. METER

the bottom line will show:

"RESET" TO CHG

the bottom line will alternate every two seconds with:

MAX HOLD

if the 580B is in the Max Hold mode. Pressing the RESET switch will cause the 580B to show:

MAX HOLD
+ = USE/ - = NO

if the +/INC switch is pressed then the Max Hold mode will be selected. If the -/CRSR switch is pressed then the Concentration meter normal mode will be selected. In either case the 580B will then return to the previous screen.

2.4.2 AUTO LOGGING SELECTION

The 580B can be configured to automatically log data points. The top line of the display will show:

AUTO LOGGING

The bottom line will alternate between "RESET TO CHG." and "ON" or "OFF". Pressing the RESET switch will cause the 580B to show:

AUTO LOGGING
+/ON -/OFF

Pressing the -/CRSR switch will turn auto logging off and return operation to the previous screen. Pressing the +/INC switch will enable auto logging and allow setting of the logging interval. The display will show:

INTERVAL 00:01
"RESET"WHEN DONE

The +/INC switch will increment the number above the cursor and the -/CRSR switch will move the cursor. The logging interval format is MM:SS (where M is minute and S is second). Pressing the RESET switch will return operation to the first auto logging screen.

2.4.3 LOCATION MODE SELECTION

The 580B may be configured to accept a six digit location code which is entered via the keypad. There is an alternate method for entering location codes however UL approval has not yet been obtained for this option. For updated information contact Thermo Environmental Instruments inc.

The display shows the currently selected location mode. For example the display will show:

Loc. code mode
"reset" to chg.

When the 580B is configured to enable operator editing of the location code, pressing the RESET switch causes the 580 to show:

Bar code mode
"reset" to chg.

The 580B is now configured for the alternate location mode (which is not presently available for use in hazardous locations). Pressing the reset switch will cause the 580B to be configured for location code mode.

2.4.4 AVERAGE TIME SELECTION

The 580B can be configured to display the average PPM from once a second up to once every four minutes. The display will show:

AVERAGE = 0:01
"RESET" TO CHG

Pressing the RESET switch will cause the 580B to show:

AVERAGE = 0:01
"RESET"WHEN DONE

The +/INC switch will increment the number above the cursor and the -/CRSR switch will move the cursor. The average time format is M:SS (where M is minutes and S is seconds).

NOTE: The maximum averaging interval is four minutes.

2.4.5 ALARM SETTING

The 580B will display the current alarm setting on the top line of the display. The setting may be changed by simultaneously pressing the RESET switch with either the +/INC switch to increment the digit above the cursor or the -/CRSR switch to move the cursor.

2.4.6 LAMP SELECTION

The 580B will display:

LAMP

on the top line. The bottom line will alternate every two seconds between:

"RESET" TO CHG

and the currently selected lamp setting and its associated serial number.
i.e.

11.8eV 000000

By pressing the RESET switch, the 580B will display:

+/10eV -/11eV

on the bottom line. Pressing the +/INC switch will select the 10.0 eV lamp. Pressing the -/CRSR switch will select the 11.8eV lamp. In either case the 580B will then allow editing of the lamp serial number. The display will show:

SERIAL # 000000
"RESET"WHEN DONE

The +/INC switch will increment the number above the cursor and the -/CRSR switch will move the cursor. Pressing the RESET switch will return operation to the original lamp screen.
When using a 10.0 eV lamp or a 10.6 eV setting should be selected. When using an 11.8 eV lamp the 11 eV setting should be selected.

2.4.7 RESPONSE FACTOR SETTING

The current Response Factor setting will be displayed on the top line of the display. The Response Factor may be changed by simultaneously pressing the RESET switch with either the +/INC switch to increment the digit above the cursor or the -/CRSR switch to move the cursor.

The response factor is used to equate the response of one organic vapor with that of the calibration gas. The current reading is always multiplied by the response factor in order to obtain the displayed concentration. A response factor of one will not change the displayed concentration.

2.4.8 CALIBRATION

The 580B will display:

"RESET" TO
CALIBRATE

The calibration mode may be entered by pressing the RESET switch.
The 580B will display:

RESTORE BACKUP
+ = YES

The previous calibration information may be restored by pressing the +/INC switch. The 580B will then return to the previous screen. If the backup is not desired, by pressing the -/INC switch the calibration routine will continue. The display will show:

ZERO GAS
RESET WHEN READY

Once zero gas has been introduced the RESET switch should be pressed. The 580B will then zero the instrument. The 580B will display:

MODEL 580B
ZEROING

Once the 580B has been zeroed the 580B will display:

SPAN PPM = 0000

The Span gas concentration may now be entered by simultaneously pressing the RESET switch and either the +/INC switch to increment the digit above the cursor or the -/CRSR switch to move the cursor. Once the span gas concentration has been entered the +/INC switch should be pressed.

The 580B will then display:

SPAN GAS
RESET WHEN READY

Once the span gas has been introduced the RESET switch should be pressed. The 580B will then calibrate the instrument. The 580B will display:

MODEL 580B
CALIBRATING

Once the 580B has been calibrated the 580B will go back to the beginning and display:

"RESET" TO
CALIBRATE

If during the zeroing or calibrating of the 580B a steady reading was not seen then the 580B will display:

CAL ERROR
RESET WHEN READY

Pressing the RESET switch will return the 580B to zeroing or calibrating (depending of course on which it came from).

See Section 4.1 for tips on calibrating the 580B.

2.4.9 FREE SPACE INDICATION

This section will give a rough indication of how much room is left for logging data points. The screen will display a bar graph on the top line and the amount of free space on the bottom line. The number indicates the total number of bytes which are available. Each data point takes fifteen bytes. Other bytes may also be needed in order to store other important information. This is why only a rough indication of room may be given.

2.5 ACCESS MODE

The Access mode is entered by pressing the +/INC switch from the main menu. The 580B has four access levels, zero through three. Level zero will only allow the operator to log data points and of course to change access levels (only if the access code is known). Level one will also allow the user to change the user identification number. Level two will allow the user complete access to the Parameters mode, and allow viewing of the date and time. Access level three allows complete access.

The access mode has three sections:

1. Access level
2. User identification number
3. Instrument number

Pressing the +/INC switch will advance the 580B to the next section. Pressing the -/CRSR switch will advance the 580B to the previous section.

TABLE OF ACCESS LEVELS

ACCESS LEVEL	OPERATIONS ALLOWED
0	Change access level Log data
1	All above operations View time and date View communication format Display logged data Change user I.D.
2	All above operations Change operating Parameters Reset logged data
3	All operations available

2.5.1 ACCESS LEVEL

The screen will display:

ACCESS LEVEL 3
"RESET" TO CHG

By pressing the RESET switch the 580B will display:

KEY 00003
"RESET" WHEN DONE

Please note that in both screens the 3 indicates the current access level and may not necessarily be a three.

In order to change the access level the +/INC switch may be pressed to increment the digit above the cursor and the -/CRSR switch may be pressed to move the cursor. The desired access level should be entered in the right most digit. Note that only access levels between zero and three are legal. The remaining four digits are the access code. The access code will be 0000 when the instrument is shipped. The access code should then be entered. Once this is done press the RESET switch. The 580B will then return to the previous screen.

If the access code entered was not the proper access code, or if the access level was not a legal access level then the access level will not be changed.

The last and most important point regarding the access level is how to change the access code. The access code is the four rightmost digits of the instrument number. The instrument number is only viewable (and therefore only changeable) while in access level three.

2.5.2 USER IDENTIFICATION NUMBER

The screen will display:

I.D.# 014563977
"RESET" TO CHG

By pressing the RESET switch the 580B will display:

I.D.# 014563977
"RESET" WHEN DONE

The user identification number may be changed by pressing the +/INC switch to increment the digit above the cursor and the -/CRSR switch to move the cursor. The user identification number is a nine digit number (just right for fitting a social security number). Once the user identification number has been entered press the RESET switch and the 580B will return to the previous screen.

2.5.3 INSTRUMENT NUMBER

The screen will display:

INSTR # 000000
"RESET" TO CHG

By pressing the RESET switch the 580B will display:

INSTR # 000000
"RESET" WHEN DONE

The instrument number may be changed by pressing the +/INC switch to increment the digit above the cursor and the -/CRSR switch to move the cursor. Once the instrument number has been entered the RESET switch should be pressed. The 580B will then display the previous screen.

When the instrument number is changed it is very important that the last four digits be remembered. These digits are the access code and therefore will need to be known in order to change the access level.

2.6 CLOCK MODE

The Clock mode is entered from the Main menu by pressing the SPKR switch. The screen will display the date and time on the top line. The bottom line will display:

"RESET" TO CHG

By pressing the RESET switch the 580B will display:

"RESET" WHEN DONE

The date and time may be changed by pressing the +/INC switch to increment the number (or in the case of the month the months abbreviation) above the cursor. The -/CRSR switch will move the cursor. Once the proper month has been entered the RESET switch should be pressed. The 580B will return to the previous screen.

The date and time will be maintained even when the instrument is turned off! It is however advisable that the date and time periodically be checked to ensure that it is correct.

2.7 COMMUNICATION MODE

The Communication mode is entered from the main menu by pressing the RESET switch. The Communications mode has four sections.

1. Communicate with printer or computer
2. Display logged data
3. Reset logged data
4. Set communication parameters

Pressing the -/CRSR switch will advance the 580B to the next section.

NOTE: A detailed discussion of communication protocol is given in Appendix A. Further discussion of communication may be found in Section Seven.

2.7.1 COMMUNICATE WITH PRINTER OR COMPUTER

The 580B is capable of communicating with a computer or outputting logged data to a printer. The 580B will display:

COMMUNICATE?

"+" = YES

if the computer format is selected or it will display:

OUTPUT TO PRINTER
"+" = YES

if the printer format is selected. In either case pressing the +/INC switch will cause the 580B to try to establish communication. Pressing the -/CRSR switch instead will cause the 580B to advance to the next section.

2.7.2 DISPLAY LOGGED DATA

If at least one data point has been logged the 580B will display:

DISP. LOG DATA?
"+" = YES

By pressing the +/INC switch the 580B will display the first data point. The date and time which the data point was logged will be displayed on the top line. The bottom line will alternate between the location code and the PPM. Pressing the +/INC switch will advance to the next logged data point. This will continue until there are no more data points at which time the 580B will display:

NO DATA STORED

The MODE/STORE switch may be pressed to return to the Run mode.

2.7.3 RESET LOGGED DATA

The logged data can be erased so that more data points may be logged. The screen will display:

RESET LOG DATA?
"+" = YES

Pressing the +/INC switch will erase all of the logged data points. The 580B will then advance to the next section.

2.7.4 COMMUNICATIONS PARAMETERS

The 580B can be configured to communicate with a printer or a computer. The baud rate may also be set for 9600, 4800, 2400, 1200, 900, 600, 300, or 150 baud. The 580B will display the current communication format (computer or printer) on the top line and the current baud rate on the bottom line. Pressing the RESET switch will cause the 580B to display:

COMPUTER FORMAT
+ = USE - = NO

Pressing the +/INC switch will select the computer format and the 580B will advance to the baud rate screen (see below). Pressing the -/CRSR switch will cause the 580B to display:

PRINTER FORMAT
+ = USE - = NO

Pressing the +/INC switch will select the printer format and the 580B will advance to the baud rate screen (see below). Pressing the -/CRSR switch will cause the 580B to display the previous screen.

The baud rate screen will display the currently selected baud rate on the top line. The bottom line will display:

+ = USE - = NO

Pressing the +/INC switch will cause the displayed baud rate to be selected and the 580B to show the selected format on the top line and the baud rate on the bottom line. Pressing the -/CRSR switch instead will cause the next lowest baud rate to be displayed.

2.8 BATTERY / CHARGER

The model 580B uses a 1.2 amp hour lead acid (gel cell) battery. There is protection circuitry potted directly on top of the battery. The battery is rechargeable with the charger provided with the instrument. The charger is regulated so that there is no danger of "over charging" the battery. It is suggested that the 580B be charged over the weekend (as well as each evening) during periods of heavy usage in order to ensure maximum battery charge.

SECTION III

ROUTINE MAINTENANCE

The routine maintenance of the 580B involves the calibration of the instrument, the cleaning of the lamp window, and the maintaining of charge on the battery. The following pages give instructions for routine maintenance. Figure 3.1 illustrates the detector assembly.

3.1 LAMP INSERTION AND REMOVAL

3.1.1 REMOVAL

NOTE: The 580B must be off while removing the lamp.

In order to remove the lamp the four screws which hold the case top and bottom together must first be loosened. The case bottom should be placed flat on the table and the top placed on its side next to the bottom.

The high voltage power supply is removed next by loosening the thumb screws on each side and then pulling the power supply towards the rear of the instrument (see figure 3.1). The lamp may now be removed by loosening the lamp nut.

3.1.2 INSERTION

Insertion of the lamp is accomplished by performing the above tasks in the reverse order. The lamp should be placed flat against the o-ring and the lamp nut fastened down in order to create a proper seal. The high voltage power supply should then be inserted and the thumb screws fastened down. There are three pins protruding from the high voltage power supply which should fit snugly into connectors located beneath the detector. The lamp spring (mounted in the center of the high voltage power supply) should make contact with the lamp ring.

3.1.3 LAMP CLEANING

On occasion the lamp should be removed for cleaning. Cleaning of the lamp is accomplished by cleaning the lamp surface of the UV lamp. The procedures for cleaning the different lamps are as follows:

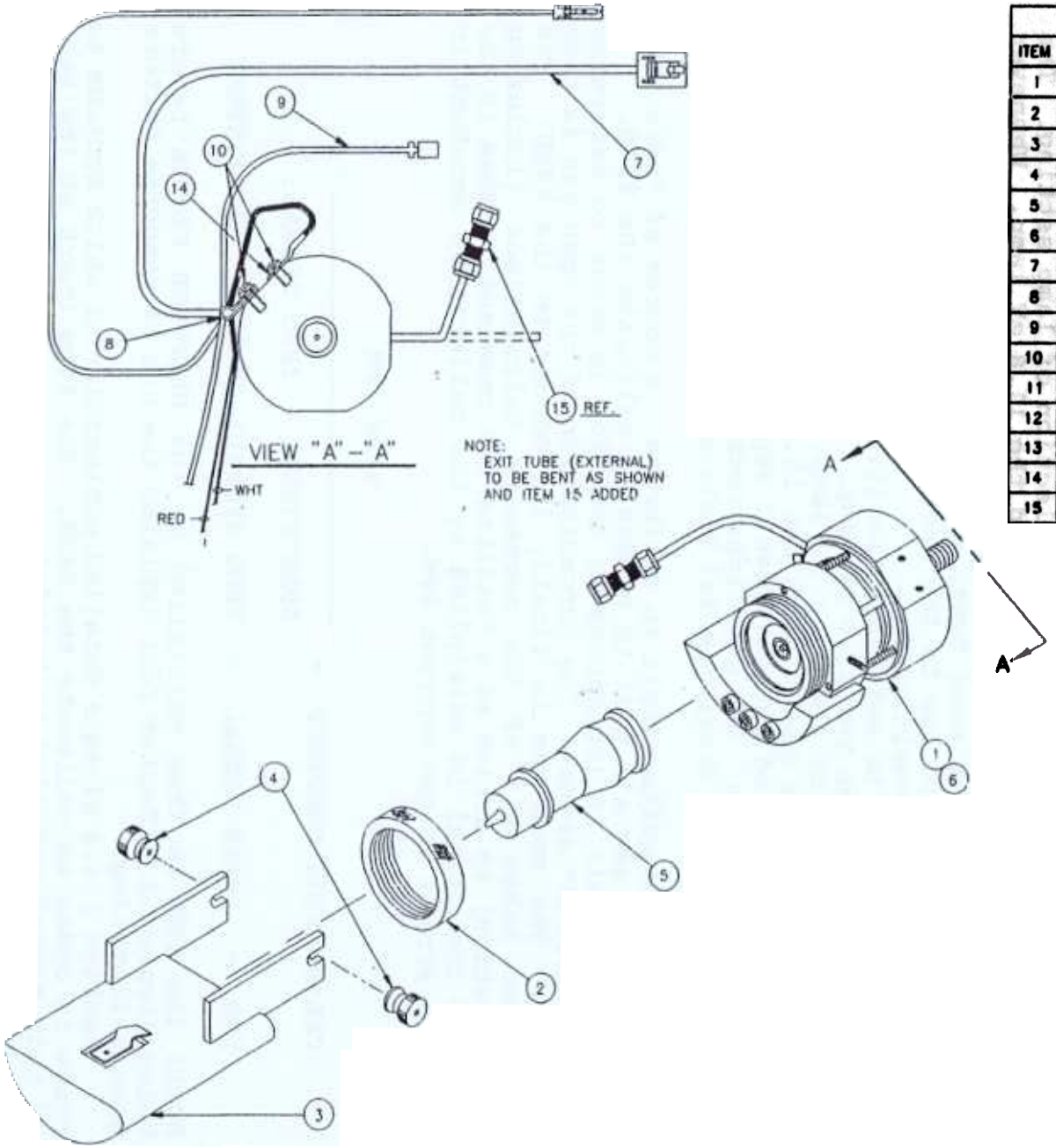
3.1.3.1 LAMP CLEANING METHOD FOR 10.6 eV OR LESS

This is accomplished by using the Aluminum Oxide scouring powder provided with the 580. First place a small amount of Aluminum Oxide scouring powder on the lens of the UV lamp. Next gently scour the lens surface with a soft tissue or cloth. Scour the lens in a rotary type motion.

After scouring the lens surface, gently blow the remaining powder from the lens. Follow this with an alcohol or acetone rinse, and then wipe dry with a soft tissue. The lamp is now able to be inserted into the detector.

3.1.3.2 LAMP CLEANING METHOD FOR 11.7 eV OR MORE

This is accomplished by gently polishing the surface of the window with anhydrous alcohol on a cotton swab, followed by an anhydrous methanol or ethanol rinse, and then wiping dry with a soft tissue. Do not allow the alcohol to remain on the surface as it will leave a film. Stubborn films may require multiple cleanings for complete removal. The lamp is now able to be inserted into the detector.



MATERIAL LIST			
ITEM	PART NO.	DESCRIPTION	QTY
1	580B-6003	DETECTOR SUB ASSY.	1
2	580S-2010	NUT - LAMP (13567)	1
3	580S-6019	PWR. SUPPLY ASSY. (13560)	1
4	12082	NUT -, KNURLED	2
5	11929	U.V. LAMP 10.0	1
6	580S-6035	DETECTOR WIRING ASSY. (13575)	1
7	580B-6001	BASE HARNESS ASSY.	1
8	4166	STRAIN RELIEF	1
9	580S-6028	SIGNAL CABLE (13568)	1
10	5814	#4-40 X 1/4" BINDER HD. SCREW	2
11			
12			
13	5510	TEFLON TUBING 1/8" 6.5" LG	1
14	5588	#4 INT. TOOTH STAR WASHER	1
15	4417	UNION-2U-316	1

580B-6004

Figure 3.1
Detector Assembly

3.2 CALIBRATION

NOTE: Chapter four should be read before calibrating the 580B in order to gain a better understanding of the concepts behind calibration of the 580B.

The following is a brief discussion of calibration as it relates to different lamps. One of the parameters in the Parameters mode (see Section 2.4) allows selection of lamp setting. The two types of lamps are the 10.0 eV and the 11.8 eV lamp. Whenever a new lamp is used the 580B must be calibrated. This is true even if the new lamp is the same type, e.g., the new and old lamp are both 10.0 eV. This is due to the fact that each lamp will have a slightly different sensitivity.

It is important to note that the 11.8 eV lamp will in general be less sensitive than the 10.0 eV lamp. This is true despite the higher energy level of the 11.8 eV lamp. The 11.8 eV lamp will however "see" certain gases which the 10.0 eV lamp will not. See Table E.1 for a list of common organic vapors and their associated ionization potentials. Any questions regarding the use of the 580B should be directed to Environmental Instruments Company Application Laboratory.

The 580B is quite simple to calibrate. A source of "zero air" and "span gas" are all that is needed to calibrate the 580B.

The zero air is introduced to the 580B in order to determine the "background" signal. The concentration of the span gas is then selected. The span gas is finally introduced to the 580B. The instrument makes all of the necessary calculations (including linearization) to arrive at a "calibration constant." When in the Run mode the signal is multiplied by the calibration constant in order to arrive at the current PPM.

$$\text{CALIBRATION CONSTANT} = \frac{\text{SPAN PPM}}{\text{SPAN SIGNAL} - \text{ZERO SIGNAL}}$$

$$\text{PPM} = (\text{SPAN SIGNAL} \quad \text{ZERO SIGNAL})\ \text{CALIBRATION CONSTANT}$$

NOTE: The PPM is then multiplied by the RESPONSE FACTOR before being displayed. Chapter four explains the use of response factors when calibrating.

Section 2.4.8 gives a detailed explanation of which buttons to press in order to calibrate the 580B. The flow chart at the back of this manual may also be helpful.

CHARGE

When there is a flashing "B" in the lower left corner of the display (while in the run mode) the battery is low. The battery is recharged by plugging the charger into the RUN/CHARGE plug at the rear of the 580B. The instrument runs while it is charging.

SECTION IV

CALIBRATION

GENERAL

The Model 580B Organic Vapor Meter is indeed a quantitative instrument and can certainly be used as such. It makes use of the Photoionization Detection System using a lamp with an ionization energy of 10.0 eV which is standard in the Model 580B. Almost all organic materials will be ionized at this energy level. There are some organic materials, such as a few of the freons, methane, ethane and propane that are not ionized and thus will not be detected. The ionization potentials for the various organic materials will simply tell whether the material will be detected by the Photoionization Detector. It does not give any clue as to the sensitivity of the detector for that particular material. Certainly, different organic vapors will have different sensitivities. It is important to understand that the Model 580B does indeed sense most organic vapors and that its response to these different organic vapors will be different.

In this section of the manual, the aspects of calibrating the Model 580B for various vapors will be discussed. In the following section discussing applications, various ways of using the features of the Model 580B will be explained along with the various methods for calibration of the 580B. There will also be applications of the Model 580B in specific instances where the organic vapors or the mixtures of organic vapors are completely unknown. The 580B can be an extremely useful tool, even in areas such as those.

FACTORY CALIBRATION TEST OF THE MODEL 580B

The Model 580B has been tested for calibration and linearity tested at the factory. The particular gas chosen for this calibration is isobutylene. The Model 580B has good response for isobutylene. Isobutylene standards prepared in air are relatively stable with time, undergoing no serious adsorption or reaction problems. The test information is included in the instrument packet. In addition to the above test a benzene standard is also run. It is important to note that the instrument was not calibrated. It was tested for calibration. Therefore, it should be calibrated by the operator before use.

4.3 METHODS OF GENERATING CONCENTRATIONS OF VARIOUS MATERIALS IN AIR

This section is not intended to be all inclusive as far as the preparation of gas and vapor standards in air are concerned. Only those methods that have been found most practical for the calibration of the 580B are discussed here. There are basically two types of standards, cylinder and bag.

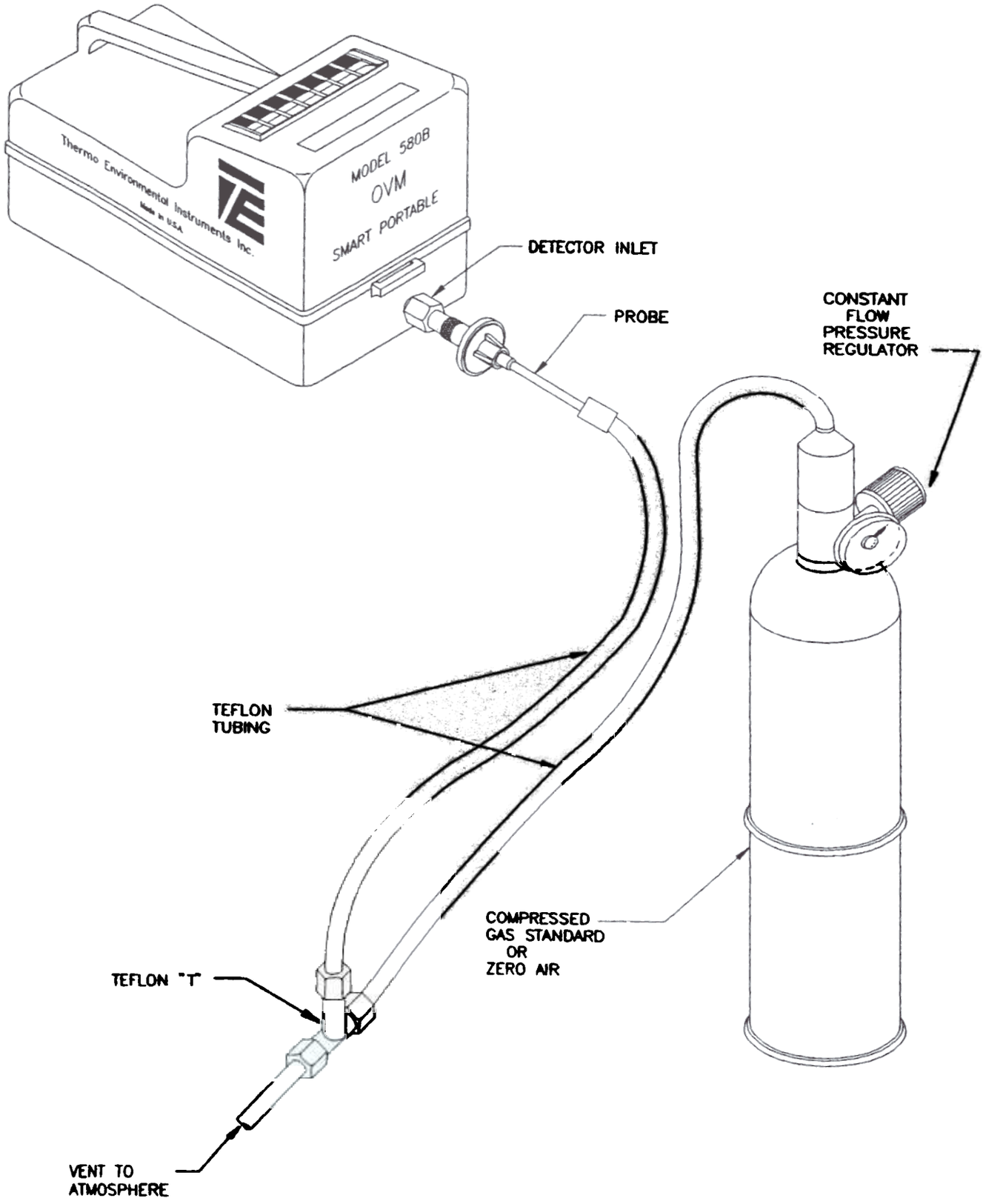


Figure 4.1
Cylinder Calibration

4.3.1 CYLINDER STANDARDS

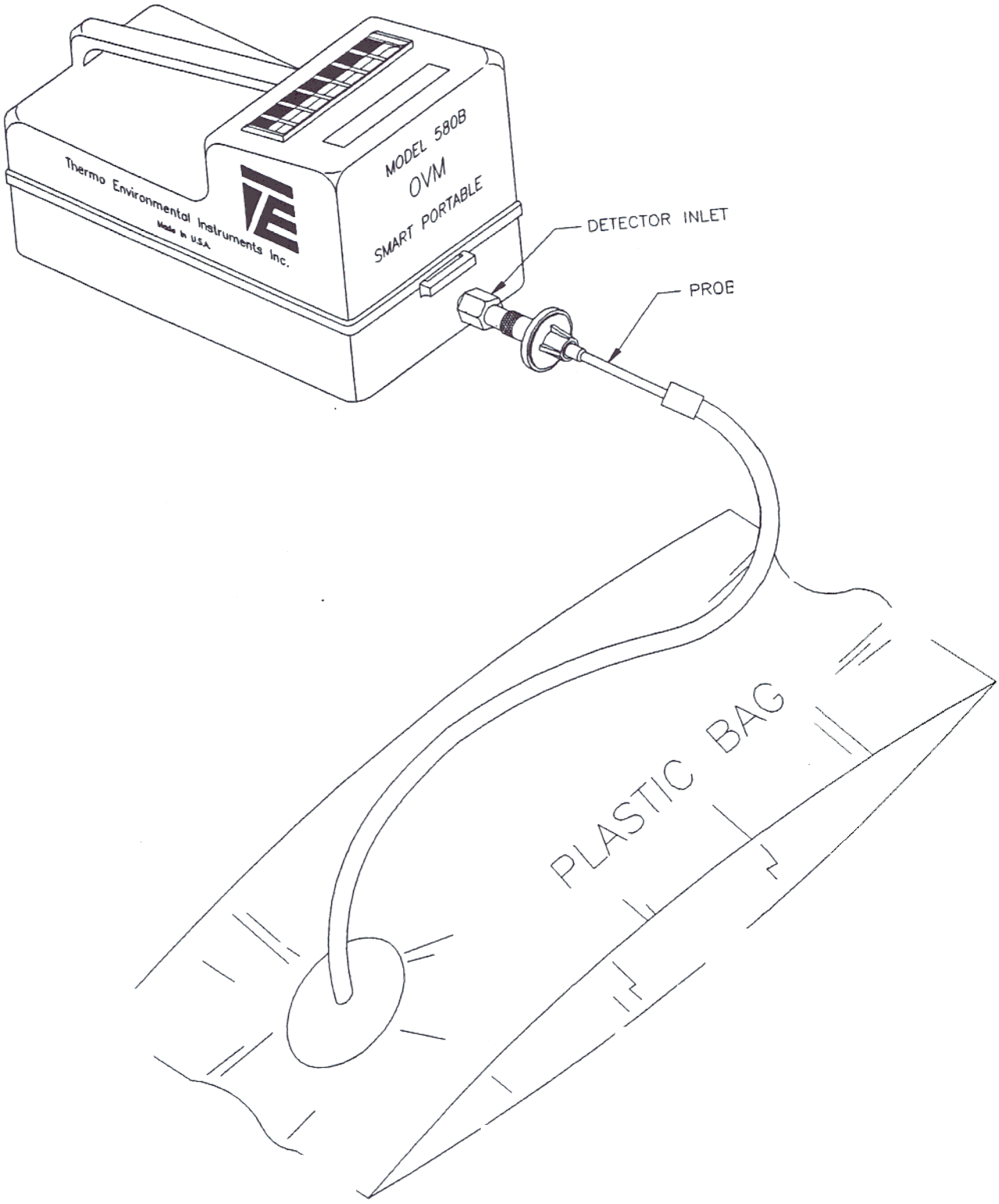
Certainly commercially available standard cylinders of gaseous materials in air offer the most convenient method of calibration. However, these are static standards. Standards prepared in this fashion in air for vapors of various organic liquids often show concentration reduction with time due to adsorption problems. In general, gases when mixed with air will maintain their concentrations with time since adsorption is generally not a problem.

However, some gases are sufficiently reactive that chemical reaction of the gas will cause a reduction of it in air. These precautions must be observed when using commercially prepared standards for calibration of the Model 580B. It is for this reason that isobutylene in air was chosen as a reference standard for factory calibration. TEI offers a cylinder standard which includes both zero and isobutylene standards. A constant flow pressure regulator sets the flow needed for calibration of the 580B. Figure 4.1 illustrates the physical calibration procedure. The inlet to the 580B is connected to the "T" as shown. It is important that this connection is tangent to the gas flow. The "T" is connected to the regulator on the standard cylinder. It is important that a length of tubing is attached to the "T" location. This prevents diffusion of ambient air into sample line. The regulator and tubing assembly will have to be moved between both the zero air and standard cylinder.

4.3.2 BAG STANDARDS (ISOBUTYLENE)

Bag standards can be prepared in a laboratory and in general are reasonable ways of calibrating the Model 580B. However, it is important that these standards be used shortly after their preparation to reduce the significance of any adsorption problems. Static standards prepared for calibration of the Model 580B are best prepared in collapsible plastic bags. This is opposed to a fixed volume container. The sampling rate of the 580B, which is 500 ml/min, requires an appreciable amount of sample. Even one minute's sampling out of a fixed container will remove 500 ml/min from it. This should not significantly reduce the pressure inside the container. Thus, the collapsible bag provides the best means as opposed to a fixed volume. A 5 gallon polyethylene bag is a convenient size to use for the preparation of static standard.

A tube is inserted into the opened end of the bag and the bag opening then sealed around the tube. The tube should have a cutoff valve or some means of closing the volume of the bag. The volume of air introduced into the bag must be measured. This is most conveniently measured by a wet test meter. However, a source of air flowing through a flow meter can be used if the flow can be held constant, then time is a measure of the volume of the air placed into the bag. All air is expelled from the bag by completely collapsing it prior to connection to the source of air.


MODEL 580B
OVM
SMART PORTABLE
Thermo Environmental Instruments Inc.
Made in U.S.A.
DETECTOR INLET
PROE
PLASTIC BAG

Fi;
ag

It can then be connected to a wet test meter or flow meter via a short length of rubber tubing hooked to the plastic tube of the bag. The air flow is started into the bag at a rate of approximately 5L/min. A total of 10 liters is a convenient volume for a 5 gallon bag. This would mean approximately 2 minutes for filling the bag. Figure 4.2 illustrates the physical configuration needed to develop bag standards.

For gaseous samples, the trace organic will be added via a glass hypodermic syringe. The 1 cc Tuberculin syringe is a convenient size. For an isobutylene standard, the 1 cc syringe is flushed with pure isobutylene and then filled to the 1cc mark. While the air is flowing into the plastic bag, the short piece of rubber tubing is pierced by the needle from the 1 cc syringe and the plunger slowly depressed such that the 1 cc of isobutylene is added to the air flowing into the plastic bag. When 10 liters of air have been added to the plastic bag, the flow is immediately stopped and the valve on the tube or the closing clamp is applied to contain the air and isobutylene within the plastic bag. It is best at this stage of the procedure not to rely solely on the diffusion of isobutylene to form a uniform mixture inside the plastic bag. Slight kneading of the plastic bag will hasten the mixing of the isobutylene in air. The plastic tube from the bag is then connected to the probe on the Model 580B via a short length of rubber tubing and the valve on the plastic tube immediately opened. The Model 580B withdraws a sample from the bag at the sampling rate of 500 ml/min. Thus, 10 liters of sample in the bag will provide approximately 20 minutes. Certainly the calibration of the 580B can be accomplished in a shorter period of time. The concentration of isobutylene in ppm by volume will be equal to the sample size, which was 1 cc, divided by the volume of the bag in liters, which would be 10 liters, times 1000. In this particular instance, the concentration would be:

$$\text{Conc (ppm by Vol)} = \frac{\text{1cc Isobutylene} \times 1000}{\text{10 L Air}} = 100 \text{ ppm}$$

4.3.3 BAG STANDARDS (ORGANICS)

On occasion there is the need to prepare standards other than the normal calibration standard. As mentioned previously, isobutylene was chosen as a standard because of its stability. If other standards are to be used, it is best to develop a relation of the other standard to a standard of known stability like isobutylene. If this procedure is followed, a response factor can be developed by comparing the other organic standard to isobutylene this technique will be discussed in a later section. The following is a suggested technique for preparing other standards.

For organic materials, which are normally liquids at room temperature, the procedure is essentially the same except that an extremely small liquid sample is injected into the flowing air stream rather than the gas sample. This technique works well

only for relatively volatile organic materials. The flowing air stream must vaporize all of the material or the calculation will be off. If the material is not rapidly volatile in that flowing air stream, the liquid should be injected through the surface of the plastic bag. Immediately after withdrawing the needle, the hole in the plastic bag should be covered with a piece of plastic tape.

Again, significant kneading of the bag will hasten the evaporation of the sample and mixing of the vapor into the air to provide homogeneous samples. The introduction of this sample into the 580B is the same as before. The calculation of the concentration of the vapor in air is a two-step procedure whereby the small volume of liquid injected into the air stream and into the plastic bag is converted to a volume of vapor. This volume of vapor is then used in the same manner as the volume of gas in the case of isobutylene. The following equations apply:

$$\text{Volume Vapor (uL)} = \frac{\text{Liquid Volume (ul)} \times \text{Liquid Density} \times 24.45}{\text{Molecular Weight}}$$

The above equation gives the vapor volume at atmospheric pressure (760 torr) and 25° C (77F).

Then:

$$\text{Concentration (ppm by Volume)} = \frac{\text{Vapor Volume (ul} \times 1000}{\text{Air Volume (liters)}}$$

The following is a sample calculation for benzene:

Liquid Volume = 2 ul

Benzene Density = 0.879 g/cc

Molecular Weight Benzene = 78.1

Air Volume = 10 Liters

$$\text{Vapor Volume} = \frac{2 \times 0.879 \times 24.45 = 0.55 \text{ ul}}{78.1} \quad \text{Benzene Vapor}$$

$$\text{Conc} = \frac{0.55 \times 1000}{10} = 55 \text{ ppm (vol}$$

The syringe used for the measurement of liquids in this particular instance is a small volume-type such as those manufactured by the Hamilton Company. A convenient size syringe is the 10 microliter volume.

4.4 580B CALIBRATION

The following procedure is applicable for both Cylinder and Bag Standards. The sequence requires both Zero gas and Span gas to be used. Span gas can be either contained as a cylinder or bag, in either case the exact concentration used must be known. This concentration will be entered to the 580 when the program provides its entry. With respect to Zero gas, there are several choices. Obviously a certified zero air standard in a cylinder presents no problem. Another choice would be to build a zero air standard in a bag. This can be simply accomplished with the set-up in Figures 4.1 and 4.2 using a charcoal scrubber to remove all the hydrocarbons present in the air. Charcoal does not absorb Methane; this does not cause a problem because the PID does not respond to it. Another approach which could be used in an emergency is to use room air unscrubbed.

This is acceptable if you know that there are no hydrocarbons present or they are exceptionally low in concentration. However, it is not recommended as a standard practice. The physical set up for cylinder calibration is illustrated in Figure 4.1; bag calibration in Figure 4.2.

4.4.1 CALIBRATION ROUTINE

(A) Set-up calibration assembly with zero air cylinder or bag as described in Figures 4.1 and 4.2.
(B) Model 580B set-up and zero calibration.

1. Power-up instrument using power plug.
2. Depress ON/OFF Key to ignite lamp and initiate sample pump.
3. Depress MODE/STORE Key.
4. Depress-/CRSR Key in response to LOG THIS VALUE? Prompt.
5. Depress-/CRSR Key to select Parameters Mode from the Main Menu.
6. Depress +/INC Key to advance thru the Run Mode selection parameter prompt.
7. Depress +/INC Key to advance thru the Auto Logging Mode selection parameter prompt.
8. Depress +/INC Key to advance thru the Average Time selection parameter prompt.
9. Depress +/INC Key to advance thru the Alarm Setting parameter prompt.
10. Depress +/INC Key to advance thru Lamp Selection parameter prompt.
11. Depress +/INC Key to advance thru Response Factor Setting parameter prompt.
12. Depress RESET Key to initiate calibration sequence.
13. Depress-/CRSR Key to decline restoration of the backup calibration.
14. Connect outlet of calibration tubing assembly to the Model 580B Detector Inlet as illustrated in Figure 4.2.
15. Introduce Zero Air to Model 580B by opening flow regulator.

16. Depress RESET Key to "Zero" Model 580B.
17. Close Flow Regulator.

C) Span Calibration - assuming that the Span gas has a concentration of 250 ppm isobutylene the following procedure is followed:

18. Simultaneously Depress RESET and -/CRSR Keys to activate the movable cursor.
19. Repeat Step 18 until the cursor is at the ones place.
20. Simultaneously Depress RESET and +/INC Keys to increment the ones place value.
21. Repeat Step 20 until the ones place value reads 0.
22. Repeat Step 18 to move cursor to the tens place.
23. Repeat Step 20 until the tens place value reads 5.
24. Repeat Step 18 to move the cursor to the hundreds place.
25. Repeat Step 20 until the hundreds place value reads 2.
26. Repeat Step 18 to move the cursor to the thousands place.
27. Repeat Step 20 until the thousands place value reads 0.
28. The LCD should now read:

SPAN PPM = 0250
"+" TO CONTINUE

29. Depress +/INC to accept the span conc. value.
30. Connect isobutylene cylinder (250 ppm) to calibration tubing assembly.
31. Connect outlet of calibration tubing assembly to the Model 580B Detector Inlet.
32. Introduce isobutylene standard to Model 580B by opening flow regulator.
33. Reset key to "CALIBRATE" Model 580B.
34. Close Flow Regulator.
35. Depress +/INC. Key in response to "RESET" TO CALIBRATE message.
36. Depress MODE/STORE to return to the Run Mode.

The instrument has been calibrated and is ready to make measurements.

4.5 DETERMINATION OF RESPONSE FACTORS

As mentioned above, the Model 580 can be calibrated with isobutylene but be set to read correctly, the concentration of another substance. This is done by using the Response Factor that is set in the parameter routine. The default for the response factor is 1.0. The Response Factor is the number that is multiplied by the measured concentration to obtain the correct concentration of the measured component. If the chemical to be measured is less sensitive on a PID than the standard, (usually isobutylene) then the Response Factor is greater than 1.0. If it is more sensitive than the standard then the Response Factor is less than 1.0.

The reason for a Response Factor is practicality. If it is

know that the sample to be measured contains only benzene and therefore the user would like to read benzene concentration directly, there are two approaches. The user could make a bag standard daily of benzene vapor in air and calibrate the 580 directly. Or the Response Factor could be used. In the latter case a bag with benzene is made only once for comparison to a cylinder of a stable standard (such as isobutylene). Then daily, the Model 580 is calibrated with the cylinder standard, a simple operation compared to the work of preparing a bag standard.

As an example, if the bag containing 55 ppm benzene in air as prepared above were measured in a 580 calibrated against isobutylene, the concentration might have been read as 91 ppm. thus the 580 is more sensitive for benzene than for isobutylene.

The Response Factor can now be calculated as:

$$\text{Response Factor (RF)} = \frac{\text{Factor STD Concentration}}{\text{580 Reading of Factor STD}}$$

$$RF = 55/91 = 0.604$$

When 0.60 is entered into the 580 as the Response Factor, the 580 will read 55 ppm for the bag.

Now the 580 need only be calibrated using an isobutylene standard and a Response Factor of 0.60 to correctly respond to benzene.

SECTION V

APPLICATIONS

5.1 GENERAL

This section discusses six applications which were done on the old model 580. These applications are discussed as they relate to the model 580B. The following applications of the Model 580B are given to show some different uses and means of calibration of the Model 580B in various practical applications. It is certainly not intended to be an exhaustive list of the uses of the Model 580B. In each situation, the stress is placed on the means of calibration and the interpretation of the readout of the Model 580B. Since the Photoionization Detector responds to virtually all organic materials and since its response varies for the different organic materials, questions can certainly arise as to just how the numbers presented on the digital display relate to anything meaningful. These applications will hopefully illustrate several ways in which these numbers can be quantitative and also illustrate uses of the 580B where accurate quantitation may be impossible.

5.2 VINYL CHLORIDE MONOMER IN REACTION VESSELS.

This particular application involved measuring the vinyl chloride content in vinyl chloride polymerization vessels following the polymerization reaction and the removal of the polymer slurry. Any residual vinyl chloride left in the reaction vessel has to be flushed and scrubbed prior to the opening of the vessel. The vinyl chloride content must be below a certain prescribed level prior to this opening. The reaction vessel is flushed with nitrogen to remove the vinyl chloride from the vessel and purge it through the filter media which remove the vinyl chloride from the nitrogen stream for recovery. During this particular operation, it is known that vinyl chloride monomer comprises significantly more than 90% of the entire organic material. In this instance, if the Model 580B is calibrated for vinyl chloride measurement, indeed the readout will be virtually the true vinyl chloride concentration inside the reactor vessel. The nitrogen exit stream prior to the vinyl chloride recovery was the point used for the analysis.

Since the plant was a considerable distance from the laboratory and since the study would require a significant period of time encompassing several weeks, it was decided to calibrate the Model 580B with the isobutylene reference standard and determine a response factor setting for a vinyl chloride standard in the laboratory.

With the response factor set at 1.0, the instrument was calibrated with isobutylene. The Model 580B was then presented with a known concentration of vinyl chloride monomer in nitrogen. The response factor for the vinyl chloride was then set in order for the Model 580B to read the correct concentration of vinyl chloride in the nitrogen. Static

standards of vinyl chloride are very definitely not stable with time due to the reaction of the vinyl chloride with itself. Thus, standards need to be prepared fresh each time vinyl chloride is to be used to calibrate an instrument. Since bag preparation, which was the technique used for this laboratory calibration of the 580B, would have been inprac tical at the plant; the use of a stable reference standard of isobutylene was chosen. Thus, at the plant site, the Model 580B could be calibrated using the isobutylene standard from a cylinder. This of course, greatly simplified the plant use of the Model 580B. This relationship to a reference standard reduces the time and equipment required at the plant such that the survey of all of the reactor vessels was completed in a short period of time with the items established for the nitrogen flush of the reactor vessels prior to opening the reactor vessels.

It is important to note that when the response factor setting was determined in the laboratory, nitrogen was used as the matrix for the bag preparation of the vinyl chloride standard. If air were used a different setting (higher) would be obtained. Since the sample was in a nitrogen matrix so should be the standard. Note also it is not necessary to have the isobutylene standard in nitrogen. In addition to correcting for differences in response between isobutylene and vinyl chloride, the response factor setting can also adjust for the different readings in nitrogen and air.

5.3 MONITORING ISOLATED PLANT AREAS FOR TOLUENE AND METHYL ISOBUTYL KETONE.

Two areas of an extensive plant operation were required to be monitored for the levels of methyl isobutyl ketone and toluene. Both of these areas were relatively isolated. In one area, methyl isobutyl ketone was the only solvent to which the atmosphere was exposed other than the potential leaks that might occur in process equipment in that same area. There were no other known solvents in use in that area and the ventilating system in effect isolated this area from other areas in the plant. In the second area, toluene had just very recently been substituted as a solvent in place of benzene due to the lower TLV for benzene. Average workplace levels were therefore needed for the toluene concentration in this work area. Again, toluene was the only solvent in this area and there was no other process equipment in the immediate area for even possible leak problems.

Notice that in both of these areas in the plant, it is certainly known from the processes occurring in that area and its relative isolation from the other areas in the plant, exactly which organic vapors will be by far the predominant vapors in the workplace air. In many instances, by simply knowing the processes involved and the chemicals in use in those processes, the qualitative aspects of the environment can indeed be established without the use of instrumentation. This is one of the most overlooked aspects in establishing what organic vapors are present in the environment. It simply involves determining what are the possible organic vapors that can be present. In general, this narrows it to several and in many cases, a single organic

vapor.

In these cases, the Model 580B can be calibrated specifically for these materials and will provide quantitative data on the levels of these materials in the workplace environment. In this particular instance, even though the laboratory to be used for the calibration of the Model 580B was at the plant site, it was desired to use a single 580B to monitor both work areas sequentially and several times throughout the course of a single day. This was to be done over a period of time to establish the variations of both methyl isobutyl ketone and the toluene in these work areas. In this particular instance, changing the response factor setting can avoid considerable calibration changes, as one moves from determining concentrations of methyl isobutyl ketone to the area where one is measuring the concentrations of the toluene vapor.

For calibration, the Model 580B response factor was set at 1.0 and the instrument spanned properly using a known reference standard of isobutylene. The Model 580B was then presented with a flowing air stream containing toluene vapor as generated in the Thermo Electron Model 360 using a toluene diffusion tube. The response factor was then adjusted so that the readout of the Model 580B corresponded to the toluene concentration in this standard.

The Model 580B was then presented with a flowing air stream containing methyl isobutyl ketone. This also was generated via a diffusion tube in the Model 360 Standards Generator. Once again, the response factor was adjusted so that the digital display gave the correct reading for the concentration for the methyl isobutyl ketone presented to the instrument.

With the instrument then calibrated with the reference isobutylene standard and knowing the proper settings of the response factors for methyl isobutyl ketone and toluene, the Model 580B was then ready for its plant survey. The area containing the toluene was monitored for a period of time with the toluene levels as noted by the 580B being recorded.

The response factor was set for this toluene reading. The instrument was then moved directly to the methyl isobutyl ketone area and the response factor adjusted to read methyl isobutyl ketone. The 580B was then able to read directly the methyl isobutyl ketone concentration in the second area. There was the possibility of leaks in process equipment in this particular area. The area in general was surveyed. If significant changes in the reading of the 580B were observed, the 580B was used as a leak sourcing instrument as described in a later section. In this fashion, it could be determined if some of the varying concentrations in this area were indeed coming from a leak in the process equipment. During the survey of this particular area, no leaks from process equipment were observed, therefore, the readings obtained on the 580B could indeed be considered the methyl isobutyl ketone concentration in this particular area.

Throughout the survey of these two workplaces, the 580B could move back and forth rapidly due to its portability and could be, in effect, recalibrated for each of the two different vapors by the mere setting of the response factor.

5.4 PETROLEUM ETHER VAPORS IN WORKSPACE AIR.

A given workplace was using petroleum ether as a paint solvent and for cleaning purposes. It was desired to quantitate the amount of petroleum ether in the air being recirculated in this particular area. Petroleum ether is a distillation fraction from crude oil. Its boiling point is slightly lower than the boiling point of gasoline. This means that petroleum ether is not a single chemical entity, but a multitude of hydrocarbons in a certain boiling range fraction. Reasonable quantitative data can be obtained here without knowing the exact chemical composition of each hydrocarbon that composes petroleum ether . For this purpose, the Model 580B can be used to measure these vapors. The 580B is initially calibrated with the response factor set at 1.0 using a reference standard of isobutylene.

The 580B is calibrated on isobutylene. Then a bag sample is prepared, as detailed above, for the quantitation of the instrument to measure the petroleum ether. In this particular instance, the petroleum ether is injected into the bag in the same fashion that liquid samples are injected. The calculation, however, has to change slightly because the ppm on a volume basis cannot be calculated without knowing the exact chemical composition of the petroleum ether. However, in a situation such as this, one can still quantitate it on a weight basis of the solvent in air. The equations below show this calculation.

Weight Vapor (mg)=Liquid Volume (uL) x Density g/ml

$$\text{Conc (mg/m3)} = \frac{\text{Weight Vapor (mg x 1000}}{\text{Air Volume liters)}}$$

For Petroleum Ether In This Example:

Liquid Volume = 3 uL

Petroleum Ether Density = 0.66

Air Volume = 10 liters

Vapor Weight = 3 uL x 0.66 g/mL = 1.98 mg

$$\text{Conc} = \frac{1.98 \times 1000}{10} = 198 \text{ mg/m3}$$

This sample in the bag is then presented to the Model 580B and the response factor adjusted so that the digital readout on the front panel provides the proper reading in mg/m3. The setting of the response factor that is needed for this reading is noted. The Model 580B can now be used to monitor reasonably quantitatively the petroleum ether in the workplace environment. Any further calibration of the instrument can be done using the reference standard of isobutylene. This is a reasonably accurate

way of giving quantitative information on the amount of solvent in air even though the results are not reported in ppm on a volume basis.

This technique can be used in general when the solvents are a mixture of materials which in general will probably be petroleum distillation fractions. It would certainly also be used in the case of gasoline vapors in air. Notice from the equations used versus the equations for determining the ppm concentration in bag samples for pure liquids, the only real thing missing is the molecular weight of the material. It may be possible to assume an average molecular weight of the solvent mixture and actually report a ppm by volume basis.

5.5 LEAK SOURCING

In this particular instance, the Model 580B is to be used for determining the presence, or absence of leaks in a chemical process plant. The Model 580B is uniquely adapted to this particular operation due to its light weight. In this particular instance, it is not necessary to accurately attempt to quantitate the readings from the Model 580B. It will be used simply to determine presence of leaks and to locate these leaks.

The Model 580B is simply calibrated against a reference standard of isobutylene as normal. No further calibration is used. It is not necessary to know the particular chemicals flowing in the different pipes or what they are in the various reaction chambers. It is only necessary to know that these materials will have some response on the Photoionization Detector. That is, that their ionization potentials are below the energy of the lamp. The standard probe of the Model 580B, with the 580B fully operational, is then simply moved along the various pipes and reactor vessels in the chemical process.

All seals are traced clear around the seal with the end of the probe. As one approaches a leak, the concentration of the organic materials in the air being sampled by the Model 580B will increase significantly. The point of maximum reading will indicate the point of the leaks. As one moves further away from the leak, the concentration of the organics in air will certainly decrease. In this very rapid fashion, the presence of leaks can be detected and their source fairly accurately pinpointed so that the leak can be repaired.

In many instances, it is not necessarily the workplace hazards of these leaks that is important, but the economics of the chemical process itself. In this instance, as in many instances, the exact composition of the organic materials being measured is really unimportant to the successful use of the 580B in a specific application. Also the exact numbers that are displayed on the digital readout of the 580B are unimportant. It is only relative magnitudes that are important in this instance.

5.6 AFTERBURNER EFFICIENCY

In a particular coating process, the material, after it has been coated, is passed into a dryer where the solvents of the coating are removed. These solvents are then vented into a

stack. To reduce the hydrocarbon emission from this plant, an afterburner had been installed to combust the organic solvents from the coating prior to release to the atmosphere. It is important to determine the efficiency of this afterburner and to follow the efficiency of the afterburner to avoid dumping excess solvent into the atmosphere and, thus, become subject to pollution fines.

The Model 580B is ideally suited to this type of operation. Again, it will be unnecessary to know the exact chemical composition of the coating solvent. The Model 580B is simply standardized against the reference standard isobutylene in the usual fashion.

The Model 580B is then connected to sample the stack gas in the dryer prior to the afterburner, noting the steady state number displayed on the digital panel meter. The 580B is then connected to the exhaust gases from the stack following the afterburner.

Again, the steady state number, as displayed on the Model 580B, is noted.

The reading prior to the burner minus the reading after the burner divided by the reading prior to the burner times 100 gives efficiency of the afterburner in the stack. This number is quite accurate, even though the Model 580B was not calibrated specifically for the solvents or solvent mixture used in this particular coating operation. The individual readings before and after the afterburner may not have the exact quantitative relationship to the actual amount of material, but their ratio will be accurate since basically the same chemical or mixture of chemicals is being measured before and after the afterburner.

5.7 SAMPLE COLLECTION OF UNKNOWN ENVIRONMENTS

The Model 580B can also be used in areas where organics are known to be present, but perhaps the exact composition of the environment is not known. This may be due to several solvents being in the same general workplace or various separate processes occurring in that same workplace, all of which could and possibly are admitting organic vapors. In plant areas such as these, the Model 580B can still be extremely useful.

The 580B is calibrated against a reference standard of isobutylene, as mentioned above. The 580B is then used as a survey tool throughout the entire plant area. The readings are logged, especially changes in these readings. The exact numbers displayed will not, in general, be a quantitative measure of the ppm of the organic vapor since it is impossible to know what organic chemical or mixture of chemicals should be used for the calibration. When high readings are obtained on the Model 580B, an evacuated sample bag can be connected to the rear of the 580B at the sample exhaust port. This bag could be virtually identical to the type of bag used for standards preparation. The Model 580B is sampling the atmosphere at the rate of 500 ml/min. The detection system of Photoionization is a nondestructive system such that the sample that is exiting the Model 580B is indeed the same material that is giving the readings on the 580B. When the 580B is seeing high readings, this is the time the bag is con-

nected to the rear for sample collection. The bag, if the same type is used for sample preparation, can hold approximately 10 liters of air sample; which would permit a sampling time of 20 minutes.

This bag sample can then be closed on removal from the 580B and transported to a laboratory for subsequent analysis to identify the individual chemical compounds present in the sample causing the high readings and to ascertain if the workplace environment is harmful at those high readings.

The use of the Model 580B coupled with the bag collection ensures that the sample that is returned to the laboratory for analysis is a sample containing the desired organic vapors. This is assured because the bag collection is used only when the Model 580B is detecting high levels of organic vapor in the environment. This is an instance of the use of the Model 580B when the type of organic vapors are not known and it is desired to know them. The 580B has a very useful function even in these areas. It should be noted that a charcoal tube could be connected to the rear of the 580B as well as an evacuated plastic bag. The charcoal tube will pass the bulk of the sample, which is air, and adsorb the organic vapors. This charcoal tube can be returned to the lab for subsequent analysis for both a qualitative identification of the materials present as well as a quantitative measure of their levels.

SECTION VI

COLLECTION TECHNIQUES

6.1 GENERAL

As mentioned in the Application Section, it is possible to use the 580B in completely unknown areas as far as the organic vapors present are concerned and still obtain meaningful data. One of the techniques described here is the use of the 580B as a means of collecting the representative samples for further identification in the laboratory regarding the specific organics that may be present in addition to their concentrations.

Two techniques were mentioned in the section under the heading "Sample Collection of Unknown Environments". One of these techniques involves the use of a bag for collection and the other involves the use of charcoal tubes as a means of trapping organic vapors. In this section, each of these techniques will be explored in further depth as to the proper way of using the 580B to collect the samples for subsequent analysis. These collection techniques are quite useful when one is using the Model 580B simply as a survey instrument. When readings on the 580B become quite high in certain areas, it is impossible to determine the exact source of the high readings to perhaps pinpoint the specific organic chemical giving rise to the reading. One may very well want to identify what the chemical or chemical mixture is that is providing the high reading. This will have to be done with instrumentation significantly more sophisticated than the Model 580B; namely, an instrument that can provide specificity as well as qualitative identification. A Gas Chromatograph is such an instrument.

If it is desired to collect some of the air to send to a laboratory for further analysis, one needs to be sure that the proper samples are taken at the proper time. This means simply that one needs to be assured that the sample sent to the laboratory is indeed a sample that has a high concentration of organic vapor present in the sample. The 580B is used to indicate the presence of the high level organic vapors. The sample then is gathered at the exit port of the 580B when the 580B is reading high values. This assures that the sample sent to the laboratory does indeed have the high level vapors present in it. This generally simplifies the sampling technique of the environment and reduces the number of samples and, therefore, the expense needed to accurately identify the organics present and to quantitate them in a laboratory.

Two design features of the Model 580B make this type of operation possible. The first is that the detection system used in the Model 580B is the Photoionization Detector which is basically a nondestructive detector. Thus, the instrument is able to sense the organic vapor using the detector and virtually the same concentration of the same materials exits the detector as entered it. This does make it possible for the collection of the exact sample contributing to the high readings.

The second feature of the 580B that allows this sample collection is that a positive displacement pumping system is used to draw the sample into the Model 580B. It is a very simple procedure then connect to the exit of this positive displacement pump and trap the sample exiting the 580B after it has passed through the detector.

6.2 BAG SAMPLE COLLECTION.

One of the most convenient ways to sample the environmental air is to simply trap the entire air sample in a collection bag. As discussed before, the bags used for the calibration of the Model 580B, as discussed under the Calibration Section, can certainly be used for collection of the air samples. There are several precautions that must be mentioned immediately relative to the use of bag sample collection. When a bag has been filled with air that has organic vapor in the air sample, the organic vapor molecules will absorb onto the inside surface of the bag. This adsorption will begin immediately on introduction of the air into the bag. It will continue to progress with time until the vapor molecules that adsorb onto the wall of the bag are in equilibrium with the vapor molecules in the air. This equilibrium depends very strongly on the bag material and the chemical entity of the vapor itself. The ambient temperature also has some effect.

As mentioned under the Calibration Procedure, when one is preparing a known vapor concentration in a bag, the bag should be analyzed very rapidly after its preparation to ensure proper calibration of the instrument. The technique here is to use the standard prepared in this fashion as soon as possible such that the adsorption that has occurred is an absolute minimum amount. This adsorption becomes a bit more serious problem in using bags for sample collection. The first problem is simply when one is reusing the bag, one has to be sure that the sample contained in the bag previously has been completely desorbed from the wall. This, in general, can be checked by using clean air to fill a bag allowing the bag to set for a short period of time, about 1 hour, and then analyzing the air in the bag. If on using the 580 to analyze this air, it shows measurable organics, then the air in the bag should be dumped and new air introduced and allowed to set for the same period of time. There will be a reduction of organic vapor on the second go-around.

If it is still too high, this procedure is repeated until the bag shows virtually no organic vapor. The bag can be evacuated and reused.

The other problem associated with adsorption and sample collection is that the sample that is collected in the bag must be analyzed as soon as possible after collection if one is going to determine quantitatively the amount of organic vapor in that bag sample. The longer the sample stays in contact with the bag, the greater the adsorption will be of the organic vapors on the surface of the bag and, therefore, the lower the concentration of the organic vapors in the air sample.

If one is interested here in only doing a qualitative analysis of the organic vapors, that is identify what vapors are

present in the air sample, the bag certainly is a convenient way of taking the sample. If one in addition to getting the qualitative analysis desires to quantitate one or more of the specific organic vapors in the sample, the bag sample should be analyzed within an hour of taking this sample. If the bag sample cannot be analyzed this soon, it is recommended that one use the charcoal tube technique explained in the next section.

There are two considerations to be given relative to the size of the bag and, therefore, the size of the sample taken. The first consideration is the amount of sample needed by the laboratory for its analysis. If the analysis is to be done by gas chromatography directly on the air sample, in general only 1 to 5 mL of sample would be required for the analysis. Therefore, this does not become a major consideration here. If, however, other analytical techniques were to be used that would require significantly higher volumes of sample, this should be taken into account.

The other consideration is the sampling time. The Model 580B samples at the rate at which the bag attached to the exit port of the 580B will be filled. If the bag can conveniently hold 10 liters of air, this means that the sampling time can be up to 20 minutes. In general, collection techniques using the Model 580B are not intended to supply a four or eight hour integrated sample. They are used simply to help identify the materials contributing to a high concentration and possibly the analysis of individual toxic organic vapors in that particular air sample. Thus, a 20 minute limitation on sampling time should not be too severe.

Certainly larger bags could be used on the exit of the 580B, allowing up to several hours of sampling time should this be desired. The difficulty then becomes that the bags are quite large and physically become difficult to manipulate. It was recommended back in the Calibration Section that perhaps a 10 liter bag would certainly be the convenient bag for the calibration of the 580B. It would appear to be also a convenient bag for collection of the samples. For this purpose, a bag that has no adsorbed vapors on the interior surface is evacuated and closed to the atmosphere. Several of these bags could be carried in a very small container. When the Model 580B is reading high values, and it is impossible to determine the source of the high values, then a bag can be connected to the exit port of the 580B and immediately opened to accept the sample exiting the 580B. The bag is kept connected to this exit as long as the 580B is giving high readings or until the bag has reached its volume capacity. At this point, the bag is removed from the exit port of the 580B, immediately closed, and returned to the laboratory for analysis.

6.3 COLLECTION USING CHARCOAL TUBES

A technique very common in industrial hygiene-type analysis is to use a small charcoal tube as a collection device. An air sample is pulled through the charcoal tube at a known flow rate for a known period of time. This flow rate and time determine the total volume of air or total sample size. The organic vapors

in the air are adsorbed on the charcoal in the tube. These vapors are then desorbed from the charcoal by adding a known volume of desorbing solvent, usually carbon disulfide. The organics end up in the carbon disulfide. The carbon disulfide is then injected into a gas chromatograph using Flame Ionization Detection. The individual organic vapors can then be identified and quantitated.

The usual charcoal tubes that are used for this type of work contain two sections. One section has approximately 100 milligrams of charcoal and a backup section has 50 milligrams. The backup section is analyzed separately from the main section to determine if there is organic vapor breakthrough in the main section. These particular size tubes have a recommended maximum flow in the neighborhood of 250 to 300 mL/min. The exit of the Model 580 is at 500 mL/min. The most advantageous way of using a smaller charcoal tube would be to split the exit stream and pass it through two parallel charcoal tubes. This would give approximately 250 mL through each tube. For analysis purposes, the charcoal of each tube is removed and combined using double the amount of solvent that would be required for a single tube.

The amount of total air that can be passed through charcoal tubes certainly depends on the concentration of organic vapor in the air. It also depends to some extent on the particular organic vapor. In general, a total sample through the smaller charcoal tube of 10 liters is a reasonably safe number to use. Since the flow is split exiting the 580B using the smaller charcoal tubes, only 250 ml/min is going through the tube. It would take 40 minutes to accumulate 10 liters passing through each of the tubes. There are charcoal tubes available in the marketplace containing 300 milligrams of charcoal in the front section and 150 milligrams of charcoal in the rear section. These tubes have correspondingly larger diameter and can accommodate higher volumetric throughputs. One of these tubes could be hooked to the exit of the 580B without doing the split. Conceivably since it contains 3 times the amount of charcoal, a safe operating total volumetric throughput would be approximately 30 liters. This would be a full hour's operating time on the Model 580B. Again, it must be stressed that the 580B when used in the particular form, is not being used as a personnel sampler to end up with the time weighted average concentration over an eight hour period. The intent here is to identify the high level organics observed on the 580B and to quantitate them following identification to determine the safe working area.

SECTION VII

COMMUNICATION

The 580B provides a serial (as opposed to parallel) communication port. There is also a communication cable provided for easy link up to a serial printer or RS-232 port of a computer. Logged data may be "dumped" (sent through the communication port) to a serial printer. Many of the 580B parameters may be set by a remote computer by using the serial port and the 580B communication software (the software is an option, part number 580A-9014).

Note: The serial port is not to be used in a hazardous location

PRINTER

The 580B can be instructed to send all of its logged data through the serial port to a printer (or a dumb terminal). The 580B printer mode should be selected (see Section 2.7.4).The serial communication cable should then be plugged into the RS-232 port at the rear of the instrument and the other end of the cable plugged into the serial port of a printer. The 580B should finally be instructed to output to the printer (see section 2.7.1).

COMPUTER

The 580B provides capabilities for remote operation. Appendix A includes a detailed technical explanation of the 580B printer and computer interface protocol. The information in this appendix is sufficient for custom software to be developed for interfacing to the 580B. Thermo Environmental however has developed communication software which implements all of the available communication capabilities in a simple "menu driven" format. Remote communication may also be accomplished by using generic communication software package such as CrossTalk. Appendix A will be helpful if this route is taken.

NOTE: Generally, the RS-232 port on an IBM PC (or compatible) is a male connector. Since the communication cable provided with the 580B is also male, a "gender changer" (a DB-25 connector which converts from male to female) is needed.

7.3 COMMUNICATION SOFTWARE (OPTIONAL)

There is communication software available which will run on an IBM PC or compatible. The software provides the capability of obtaining or changing the 580B parameters (alarm setting, response factor, or operating mode to name a few). Logged data may be stored to disk or printed to a parallel printer. Concentrations may be read and displayed on the computer screen. There are a few operations which may not be accomplished remotely (for obvious reasons). The lamp may not be changed remotely. The lamp and pump may not be turned on from the computer either.

NOTE: The communication software will not work unless the 580B is attached via the communication cable.

7.3.1 HOW TO GUIDE FOR COMMUNICATION SOFTWARE

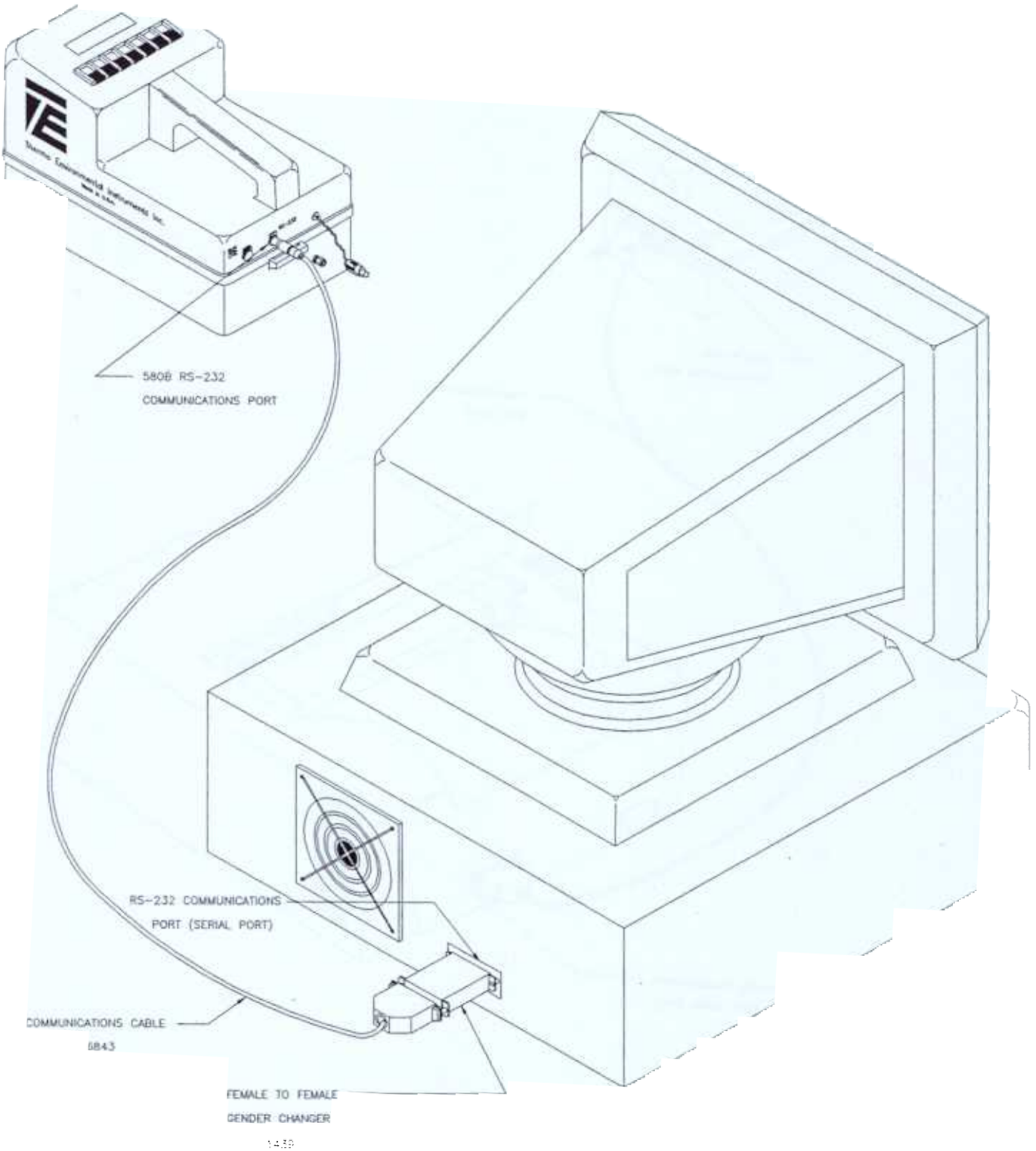
#1. The 580B must be turned on and connected to the computer's RS-232 port. The 580B must be in the computer mode (this is the default setting).

#2. The floppy disk should be inserted into the computer. Type 580B (this software was originally developed for the 580B) and then hit return. The introduction screen will appear.

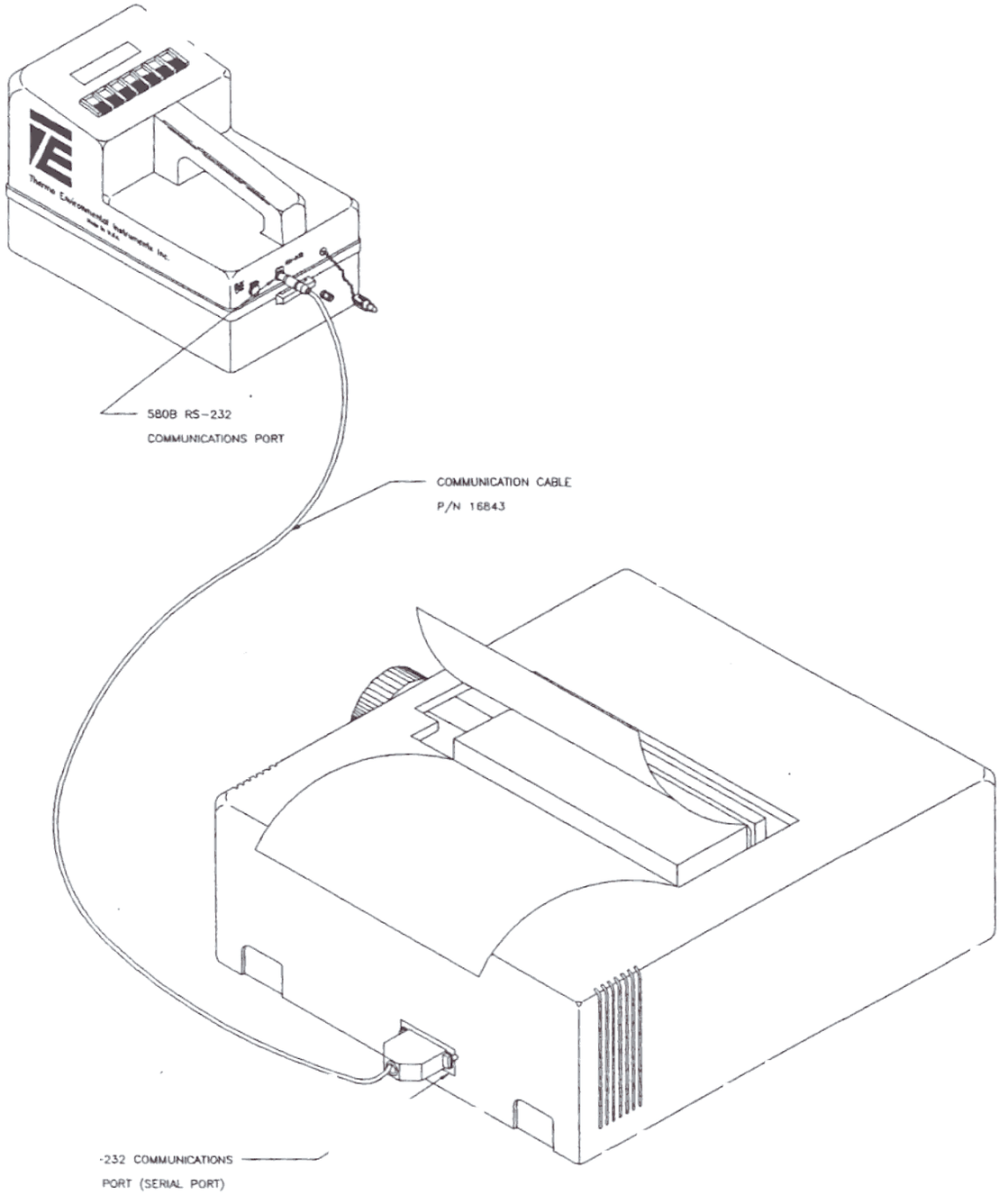
#3. The software defaults to 2400 baud (as does the 580B). If some other baud rate is desired it must match the setting on the 580B.

#4. After selection of the baud rate press return. The main menu will appear.

NOTE: If the computer's screen goes blank and the main menu does not appear, then there is a problem with the communication link. Check to be sure that the communication cable is plugged into the RS-232 port and that the 580B is on.



Fi
Se .al in Communi



Figu
Compu Communi ion

SECTION VIII

FLOW CHART

There are two flow charts which illustrate the structure of the 580B software. The first is a "Quick Start-up" flow chart. Much of the detail is not included in this flow chart in order to diagram the basic structure of the software. The second flow chart includes extensive detail of each screen and the function of the seven buttons. These flow charts provide an easy method for determining how to get at each of the many facilities provided by the 580B.

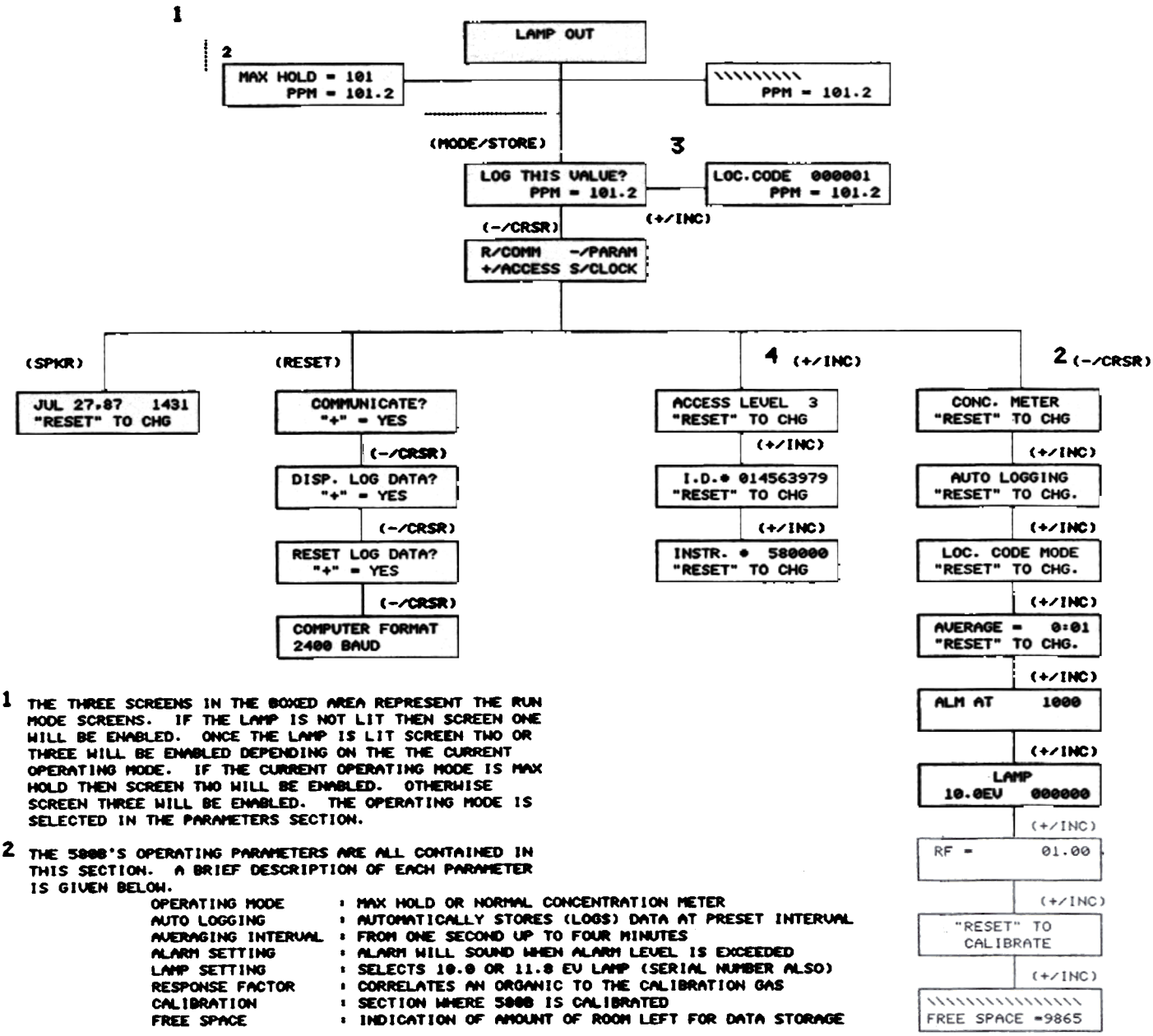
8.1 QUICK START-UP

The Quick Start-up flow chart shows each of the top level screens. The screens are ordered according to the hierarchy of the 580B software. The particular button (which advanced the 580B to the next screen) is shown in parenthesis above each screen. This flow chart does not illustrate any of the associated screens or operations (see the detailed flow chart for more in depth information).

The Quick Start-up flow chart should be fully understood before moving on to the more detailed flow chart. The best way to learn each of the flow charts is to have the 580B with you and to follow along verifying each step.

8.2 DETAILED FLOW CHART

The detailed flow chart illustrates many of the lower level screens as well as the function of buttons. Screens are shown in rectangles with the text written inside. The buttons are shown in ellipses (actually a rather flattened ellipse) with the button identifier written inside. There are a few conventions which need to be explained. The button identifiers have been abbreviated. For example the +/INC button is simply denoted as +. When two buttons need to be pressed simultaneously each identifier is shown with a slash between them. For example RESET/+ indicates that the RESET and +/INC buttons should be pressed together. Arrows indicate the direction of flow from one screen to the next.



1 THE THREE SCREENS IN THE BOXED AREA REPRESENT THE RUN MODE SCREENS. IF THE LAMP IS NOT LIT THEN SCREEN ONE WILL BE ENABLED. ONCE THE LAMP IS LIT SCREEN TWO OR THREE WILL BE ENABLED DEPENDING ON THE THE CURRENT OPERATING MODE. IF THE CURRENT OPERATING MODE IS MAX HOLD THEN SCREEN TWO WILL BE ENABLED. OTHERWISE SCREEN THREE WILL BE ENABLED. THE OPERATING MODE IS SELECTED IN THE PARAMETERS SECTION.

2 THE 580B'S OPERATING PARAMETERS ARE ALL CONTAINED IN THIS SECTION. A BRIEF DESCRIPTION OF EACH PARAMETER IS GIVEN BELOW.

OPERATING MODE	: MAX HOLD OR NORMAL CONCENTRATION METER
AUTO LOGGING	: AUTOMATICALLY STORES (LOGS) DATA AT PRESET INTERVAL
AVERAGING INTERVAL	: FROM ONE SECOND UP TO FOUR MINUTES
ALARM SETTING	: ALARM WILL SOUND WHEN ALARM LEVEL IS EXCEEDED
LAMP SETTING	: SELECTS 10.0 OR 11.8 EV LAMP (SERIAL NUMBER ALSO)
RESPONSE FACTOR	: CORRELATES AN ORGANIC TO THE CALIBRATION GAS
CALIBRATION	: SECTION WHERE 580B IS CALIBRATED
FREE SPACE	: INDICATION OF AMOUNT OF ROOM LEFT FOR DATA STORAGE

3 THESE ARE THE SCREENS USED FOR LOGGING (STORING) DATA POINTS. AFTER ENTERING THE DESIRED LOCATION CODE, SIMPLY PRESSING THE MODE/STORE SWITCH WILL LOG THE VALUE (AS WELL AS A DATE AND TIME STAMP) AND RETURN OPERATION TO THE RUN MODE.

4 THE ACCESS (SECURITY) LEVEL IS SET IN THIS SECTION. ACCESS LEVEL THREE GIVES THE OPERATOR ACCESS TO ALL OF THE 580B'S FACILITIES. LOWER ACCESS LEVEL ZERO ARE PROGRESSIVELY MORE RESTRICTIVE. ACCESS LEVEL ZERO ONLY ALLOW READING AND LOGGING OF DATA.

Figure 8.1
Quick Flow Chart

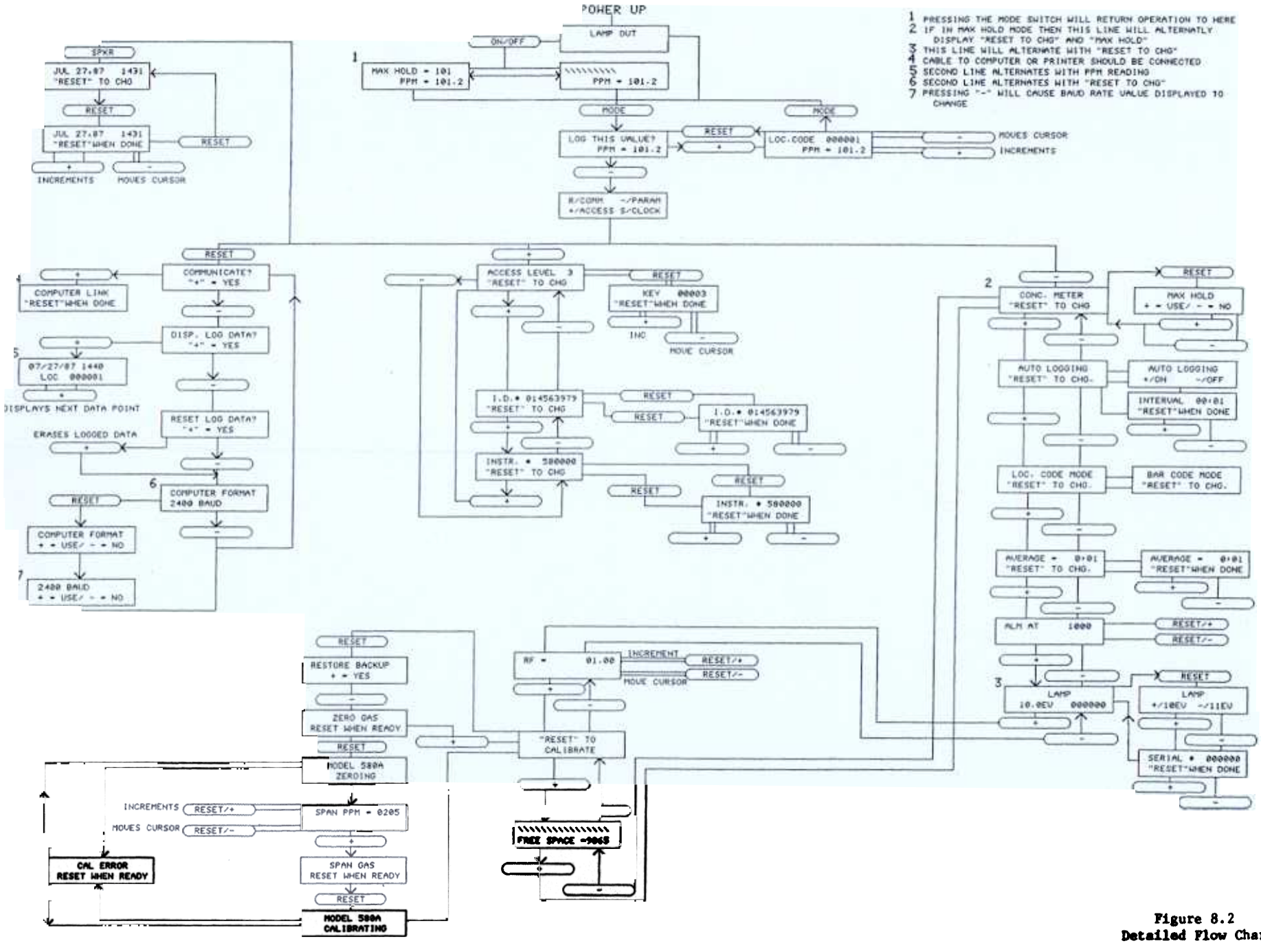


Figure 8.2
Detailed Flow Chart

APPENDIX A

COMMUNICATION

A.1 INTRODUCTION

The 580B is capable of communicating over an RS-232 link with any peripheral device which adheres to the communication protocol outlined in this document. The 580B will communicate with a peripheral device in one of two modes. While in the PRINTER mode the 580B responds to commands from its keypad and can be commanded to send characters out its RS-232 port to a peripheral device. In the COMPUTER mode the 580B responds to commands from a peripheral computer and can be commanded to send and receive data and to perform other tasks. Regardless of the mode used, the 580B sends and expects to receive data in the following format:

No Parity
1 Start Bit
8 Data Bits
1 Stop Bit
Baud Rate = 150, 300, 600, 1200, 2400, 4800 or 9600

The Baud Rate may be selected from the 580B keypad (see Section 2.7)

A.2 PRINTER MODE INTERFACE

In the PRINTER mode the 580B can be instructed to send its log data out the RS-232 port to a peripheral device such as a printer.

The 580B can be manually instructed from its keypad to output its log data. In this case the 580B sends all the log data points it has acquired thus far. The log data is saved in 580B memory and will NOT be automatically erased upon output. While in the PRINTER mode, the 580B log data file must be erased (reset) from the keypad. An example of a log data output is shown on page A-17. Notice that the 580B also sends header information which includes the following parameters: instrument number, user ID, and mode of operation. The date at the top of the header corresponds to the time when the first log data point was taken with the parameters set as shown in the header. If any of the parameters are changed and then new log data points are acquired, the 580B will send an updated header before it sends the new data points. It is also important to note that every log data point is time stamped to show when it was stored.

HARDWARE INTERFACE, PRINTER MODE

```
580B              PERIPHERAL

TxD  (B)_          )RxD
CTS  (E)(          _DTR (or "printer ready")
GND  (A)___________GND
```

The connections shown above are required before the 580B can successfully send its log data to the peripheral. An example of a cable used for PRINTER MODE communication is shown below:

SAMPLE CABLE, 580B TO HP THINKJET PRINTER

```
580B              PRINTER

TxD (B)________)RxD (3)
CTS (E)(________DTR (20)
GND (A)_________GND (1)
GND (F)_________GND (7)
```

Thinkjet connector = TRW/CINCH DB-25P plug connector

Please remember that the 580B must be placed into the PRINTER mode prior to output of log data to a printer. This is done from the 580B keypad.

HARDWARE HANDSHAKING, PRINTER MODE

The 580B will send log data out the port to a peripheral device as long as its CTS line is asserted (+V). If the peripheral has temporarily fallen behind, and consequently clears its DTR line (-V), the 580B will stop transmitting data. It will resume transmitting as soon as the peripheral reasserts (+V) its DTR line.

A.3 COMPUTER MODE INTERFACE

In the COMPUTER mode the 580B will respond to commands sent by a peripheral computer. The 580B will respond to 3 types of commands; DO, GET, and SET commands.

An example of a DO command is "DO RESET LOG (ret)" in which the 580B is instructed to reset its log which, in effect, clears all previously stored log data points.

A GET command such as "GET ALARM (ret)" allows the peripheral to change a specified parameter in the 580B to a value provided by the peripheral.

A combination of DO, SET, and GET commands allow the operator at the peripheral to perform a variety of functions needed to prepare a 580B for acquiring data.

The 580B can also be operated under "computer control". For example, the 580B can be commanded to acquire readings at specified intervals and then store the readings in a log data file.

The 580B can subsequently be commanded to send its log data to a printer, a file or the screen of the peripheral computer. This can all be accomplished through commands issued by the peripheral computer. So, effectively the 580B is under "computer control".

HARDWARE INTERFACE, COMPUTER MODE

```
580B              COMPUTER

TxD (B)__________)RxD
RxD (C)(__________TxD
CTS (E) __________DTR
                   ___DSR
GND (A)___________GND

                   ___CTS
                 !___RTS
```

The connections shown above are required before the peripheral computer can successfully communicate with the 580B. In addition, the 580B must be placed into COMPUTER mode. This is done from the 580B keypad.

An example of a cable used for COMPUTER MODE communication is shown below:

SAMPLE CABLE, 580B TO IBM PC

```
580B              COMPUTER

TxD (B)________)RxD  (3)
RxD (C)(________TxD  (2)
CTS (E)(________DTR (20)
            !___DSR  (6)

GND (A)________GND   7)
            !___GND   1)

             ___CTS  (5)
            !___RTS  (4)
```

If the peripheral's DTR line is asserted (+V), the 580B will look for and then respond to peripheral commands. As long as DTR remains high, the 580B will NOT perform functions such as counting, updating the display, storing log data, etc. If DTR is asserted, the 580B will display the following message:

"COMPUTER LINK"
"WAITING FOR COMM"

The 580B must be allowed 100ms between the time the peripheral asserts DTR and the time it sends the 580B its first message. If the 580B is busy communicating the display will read:

"COMPUTER LINK"
"COMMUNICATING"

When DTR is low (-V), the 580B will return to its normal mode of operation where it counts, updates the display, etc. and will NOT respond to peripheral commands.

XON/XOFF HANDSHAKING, COMPUTER MODE

If the 580B is transmitting log data and detects that an XOFF has been received, it will stop transmitting. The 580B will not resume transmission until the peripheral device sends XON. The 580B will respond to XON and XOFF characters only if it is NOT communicating with the computer (i.e., if 580B CTS is NOT asserted).

It is important to note that if the 580B receives an XOFF it will stop transmitting and will simply wait for the peripheral to send XON, it will not acquire data, update the display or perform other functions. The 580B is essentially "locked up" until it receives XON. For this reason, it would be good programming practice to send out an XON to the 580B prior to 580B/peripheral communication and afterwards also. Consequently, if an XOFF character is sent to the 580B inadvertently, the 580B will not be locked up indefinitely.

PERIPHERAL/580B COMMAND MESSAGES

A command message is a string of upper-case ASCII characters terminated by an ASCII carriage return. The carriage return may immediately follow the command or a space may separate the command and the carriage return as shown in the example below. The command messages which the 580B will accept from the peripheral are listed in TABLE A.1. The 580B will accept the messages as shown in TABLE 1 or the command portion of the message can be abbreviated as follows:

"S R F 01.00 (ret)" instead of
"SET RESPONSE FACTOR 01.00 (ret)"

Please note that the abbreviation must contain the first letter of each and every word of the command. There are some additional guidelines for abbreviating the SET OPERATING MODE commands.

If the abbreviated version of a command is sent, an ASCII blank must separate each letter of the command and must separate the command from the data. Note that the command message will contain data only if a SET command is being sent.

If the spelled-out version of a command is sent, an ASCII blank must separate each word of the command and must separate the command from the data.

Every SET command message contains a data value. The data sent as part of a SET command must conform to the formats described in TABLE A.1. It is important to note that the 580B does NOT perform error-checking on data sent as part of a SET command.

It is up to the user to insure that the data value is "reasonable" and formatted as shown in TABLE A.1.

The following are examples of valid command messages:

"SET ACCESS LEVEL 3 (ret)"
or "S A L 3 (ret)"

"SET REAL TIME 02/15/86 1723 (ret)"
or "S R T 02/15/86 1723 (ret)"

580B/PERIPHERAL RESPONSE MESSAGES

A response message is a string of upper-case ASCII characters terminated by an ASCII carriage return. The response messages which the 580B will send to the peripheral are listed in TABLE A.1. The messages which the 580B sends in response to a GET command contain data formatted as shown. The notes which follow TABLE A.1 describe the GET command response messages in more detail.

SOFTWARE HANDSHAKING, COMPUTER MODE

Every command message must be preceded by the "WAKE UP/PROCEED" sequence. This sequence begins when the peripheral sends a WAKE UP ("?") character to the 580B. The 580B must respond with a PROCEED ("!") character before the computer can send a command message. In the discussion to follow, the WAKE UP/PROCEED sequence will be referred to as (WAKE UP/PROCEED).

The peripheral sends command messages to the 580B an entire line at a time. When the 580B receives the command line it will echo the line back to the peripheral. The peripheral will examine the echo to determine if the 580B received the command correctly. If the echo was correct, the peripheral will signal the 580B with the PROCEED character. The 580B will then perform the task specified by the command message. In the discussion ahead, the command message and echo sequence will be referred to as (COMMAND/ECHO/PROCEED).

The software handshaking sequences for each of the 3 types of commands are given below:

DO COMMANDS

The handshake sequence for a DO command is as follows:

1. (WAKE UP/PROCEED)
2. (COMMANDS/ECHO/PROCEED)
3. 580B performs task
4. 580B sends PROCEED or ERR

If the 580B was able to successfully complete the task it will send a PROCEED character in step 4, otherwise it will send the error message ("ERR").

GET COMMANDS

The handshake sequence below applies to all of the GET commands with the exception of GET LOG DATA and GET CONTINUED LOG:

1. (WAKE UP/PROCEED)
2. (COMMAND/ECHO/PROCEED)
3. 580B sends data message
4. Peripheral echoes data message
5. 580B sends PROCEED or ERR

The message sent by the 580B in response to a GET command are shown in TABLE 1. When the peripheral receives the message containing the data it echoes the entire message back to the 580B. If the echo is correct the 580B will send the PROCEED character so that the peripheral knows it received the data correctly. If the echo is not correct, the 580B will send "ERR".

The GET LOG DATA and GET CONTINUED LOG commands differ from the other GET commands in that the 580B sends an indefinite number of data values. The handshake sequence for these commands is as follows:

1. (WAKE UP/PROCEED)
2. (COMMAND/ECHO/PROCEED)
3. 580B sends a log data point message
4. If message = "EOT" (end of transmission) then DONE, otherwise go on to step 5.
5. Peripheral echoes entire message
6. 580B sends PROCEED or ERR
7. Peripheral sends PROCEED
8. Go to step 3

In step 4, the 580B sends "EOT" if it has sent all the log data points available. If some time later the peripheral sends "GET CONTINUED LOG (ret)" the 580B will send any additional data points it may have acquired since the GET LOG DATA command. In step 6, the 580B will send PROCEED if the peripheral echoed the message correctly in step 5. The 580B will also increment its data buffer pointer. If however, the peripheral did <u>NOT</u> correctly echo the message in step 5, the 580B will send "ERR" in step 6 and will <u>NOT</u> increment its data buffer pointer. This means that the next time through step 3, the 580B will send the same data point again. In either case, the peripheral must send a PROCEED in step 7.

COMMANDS

handshaking sequence for a SET command is as follows:

1. (WAKE UP/PROCEED)
2. (COMMAND/ECHO/PROCEED)
3. 580B sets parameter to value
4. 580B sends PROCEED or ERR

In step 3 the 580B sets the parameter specified by the command to the value provided by the peripheral in the command message. The data value sent in the SET command message must be formatted as shown in TABLE 1. If the 580B is able to successfully set the parameter it will send the PROCEED character, otherwise it will send "ERR".

NOTE: The flowcharts shown in Figures A.1 - A.4 are included to further explain the software handshaking sequences required for successful communication between the peripheral and the 580B.

TABLE A.1
MESSAGE FORMATS

PERIPHERAL COMMAND	580B RESPONSE
DO COMMANDS	
DO END COMMUNICATIONS (ret	! (ret)
DO RESET LOG (ret)	! (ret)
GET COMMANDS	
GET ACCESS LEVEL (ret)	ACCESS LEVEL I (ret)
GET ALARM SETTING (ret)	ALARM SETTING IIII (ret)
GET CONTINUED LOG (ret)	(see notes which follow)
GET INSTRUMENT NUMBER (ret)	INSTRUMENT # IIIIII (ret)
GET LOCATION CODE (ret)	LOCATION CODE IIIIII (ret)
GET LOG DATA (ret)	(see notes which follow)
GET OPERATING MODE (ret)	OPERATING MODE: ASCII (50) (ret)
GET MAX READING (ret)	LAST MAX VALUE IIII ASCII (8) (ret)
GET RATEMETER READING (ret)	LAST CONC VALUE IIII ASCII (8) (ret)
GET REAL TIME (ret)	REAL TIME CLOCK II/II/II IIII (ret)
GET USER ID (ret)	USER I.D. # IIIIIIIII (ret)
GET RESPONSE FACTOR (ret)	RESPONSE FACTOR II.II (ret)
GET SPAN CONCENTRATION (ret)	SPAN CONCENTRATION IIII (ret)
GET VERSION NUMBER (ret)	580B VERSION 1.0 (ret)
GET LOGGING INTERVAL (ret)	I:II (ret)
SET COMMANDS	
SET ACCESS LEVEL I (ret)	! (ret)
SET ALARM SETTING IIII (ret)	! (ret)
SET INSTRUMENT NUMBER IIIIII (ret)	! (ret)
SET LOCATION CODE IIIIII (ret)	! (ret)
SET OPERATING MODE ASCII (50) (ret)	! (ret)
SET REAL TIME II/II/II/ IIII (ret)	! (ret)
SET USER ID IIIIIIIII (ret)	! (ret)
SET RESPONSE FACTOR II.II (ret)	! (ret)
SET SPAN CONCENTRATION IIII (ret)	! (ret)
SET LOGGING INTERVAL I:II (ret)	! (ret)

NOTES ON TABLE A.1

The peripheral will receive data back from the 580B in response to a GET command only. The 580B response to a DO or SET command is the PROCEED ("!") character. This is because the 580B does not actually return data but signals the peripheral with the PROCEED character if it was able to perform the task requested. If for some reason the 580B is unable to perform the task it will send an error message "ERR" rather than PROCEED.

The data values sent by the peripheral as part of a SET command message and the data received by the peripheral in response to a GET command must be formatted as shown in TABLE A.1.

The data format codes used in TABLE 1 are described below:

I	a single digit
III...II	an integer "string", the number of I's shown indicates the length of the string. Note: the "string" MUST be the length specified, use leading zeros if necessary.
ASCII (n)	an ASCII string with a maximum of n characters

Several of the command messages listed in TABLE 1 require additional explanation. These comments are listed according to the command name:

DO RESET LOG

This command instructs the 580B to clear its log data file All log data values acquired previously will be erased.

GET CONTINUED LOG

This command instructs the 580B to send any log data points acquired since the last GET LOG DATA command. The format of the 580B response will be the same as the response to the GET LOG DATA command.

GET LOG DATA

This command instructs the 580B to send all of its log data points. The log data file is saved in 580B memory and is NOT automatically erased upon output. The log data file may be erased (reset) with the DO RESET LOG command.

GET OPERATING MODE

The 580B responds to a GET OPERATING MODE command by sending a string "MODE: ", followed by an ASCII string which describes the current 580B mode of operation. The 580B responses to the

GET OPERATING MODE command are listed below:

OPERATING MODE: CONCENTRATION METER NORMAL
OPERATING MODE: CONCENTRATION METER MAX HOLD

GET MAX READING

The 580B responds to a GET MAX READING command by sending the max value as shown in TABLE A.1. After the 580B sends the max reading it resets the max value to 0.

GET REAL TIME

The 580B will return its real time as an ASCII string in the following format: "05/29/86 1422"

SET ACCESS LEVEL

The access level must be an integer in the range 0 to 3

SET INSTRUMENT NUMBER

The instrument number is a string of 6 integers. If the instrument number = 2 then the number must be represented as 000002 (i.e. leading zeros must fill in excess spaces). An example of a valid SET INSTRUMENT NUMBER command is "SET INSTRUMENT NUMBER 000002 (ret)".

SET LOCATION CODE

The location code is a string of 6 integers. If the location code = 234 then the correct SET LOCATION CODE command is "SET LOCATION CODE 000234 (ret)". (Leading zeros must fill excess spaces.)

SET OPERATING MODE

As shown in TABLE A.1, the command SET OPERATING MODE must be followed by an ASCII string which describes the mode. The list of valid SET OPERATING MODE commands along with valid abbreviations are listed below:

SET OPERATING MODE CONCENTRATION (ret)
S O M C (ret)

SET OPERATING MODE MAX HOLD (ret)
S O M M H (ret)

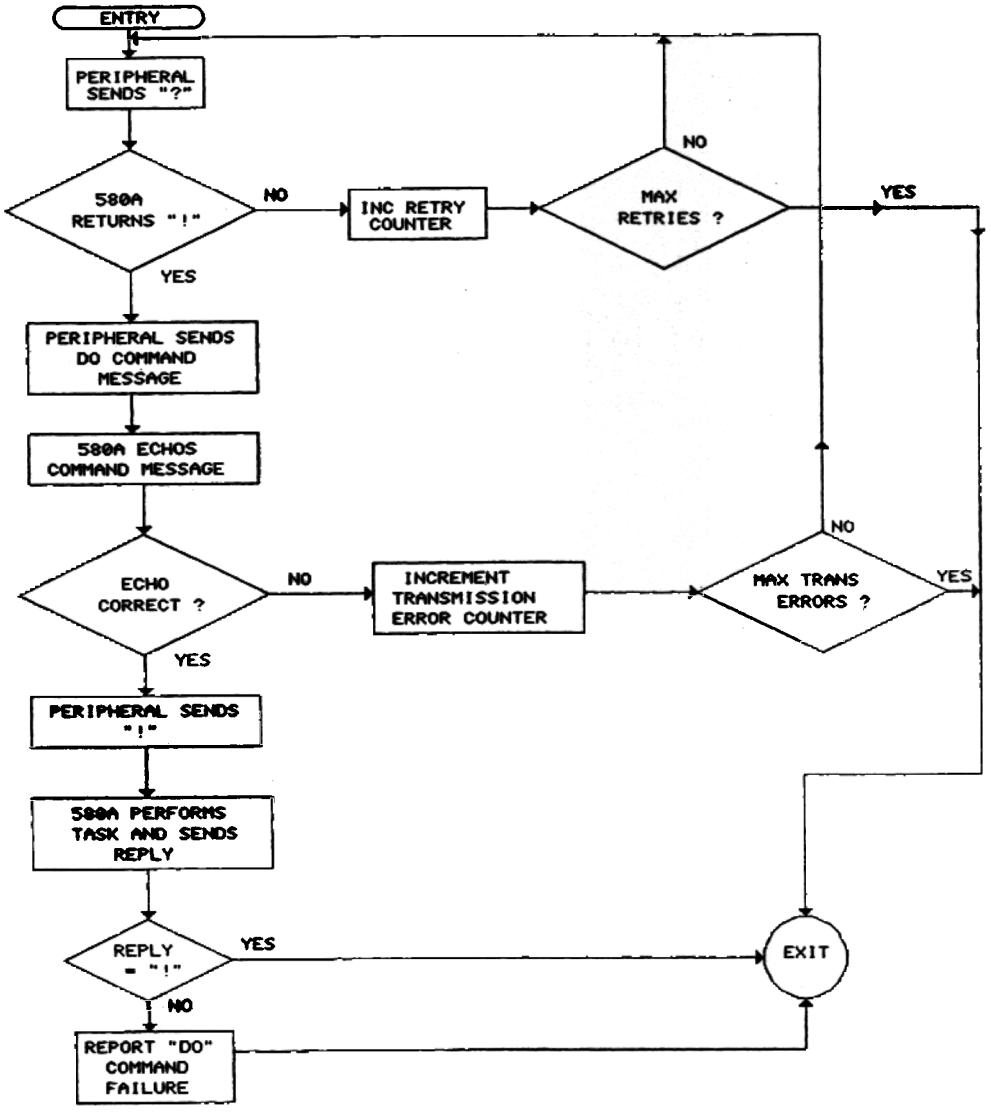


Figure A.1
Software Handshake
Do Commands

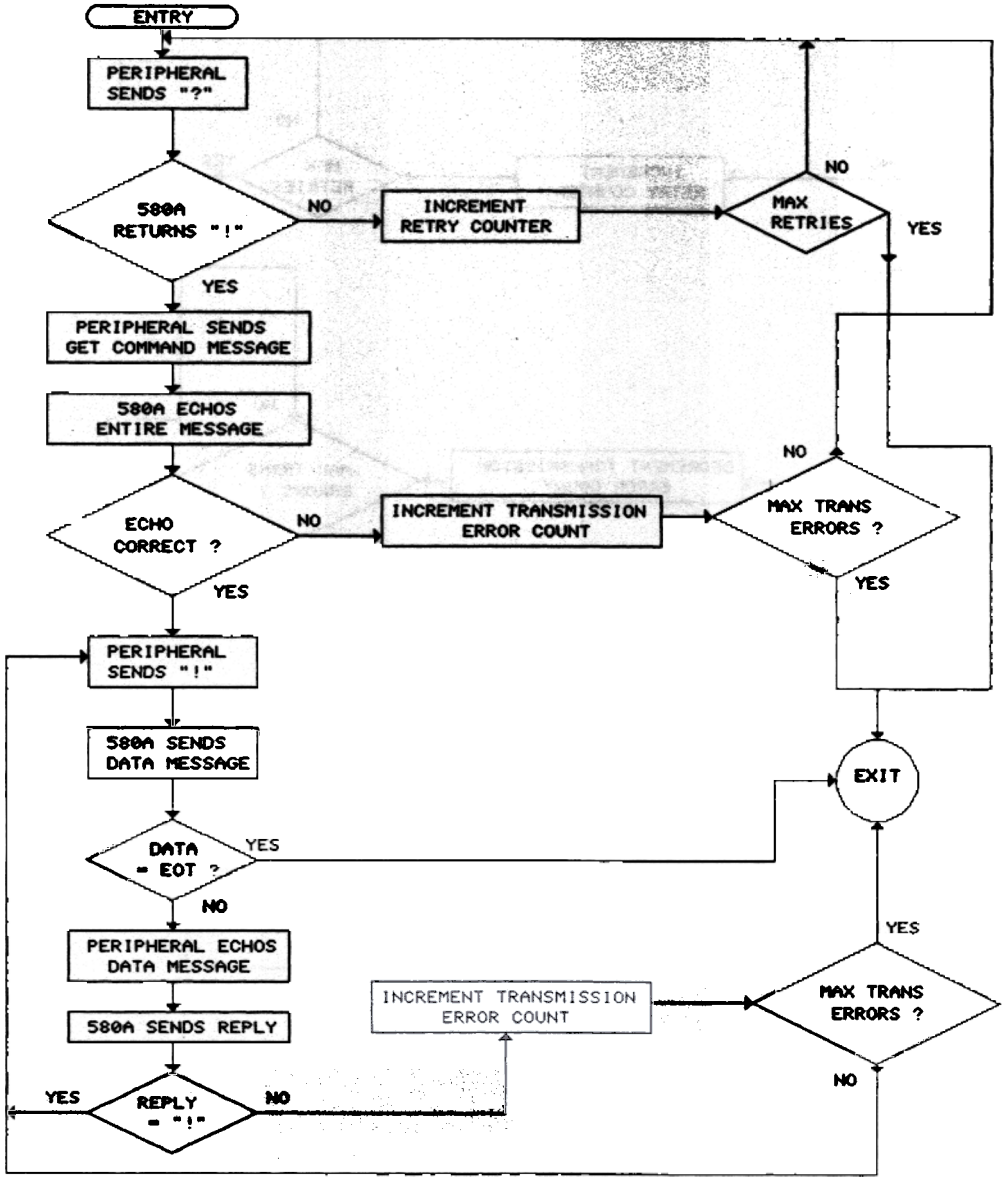


Figure A.2
Software Handshake
Get Log Data, Get Continue Log Command

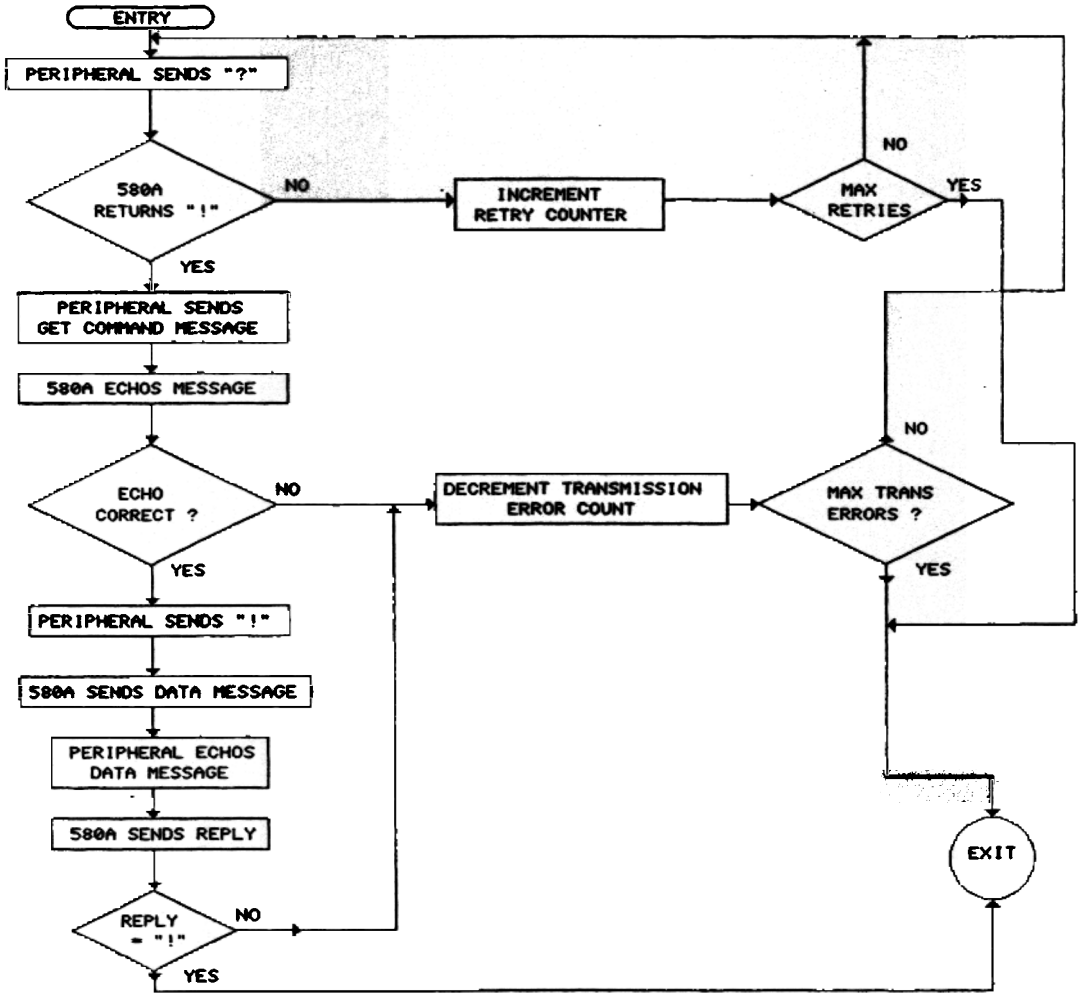


Figure A.3
Software Handshake
Get Command
(Except Get Log Data, Get Continue Log)

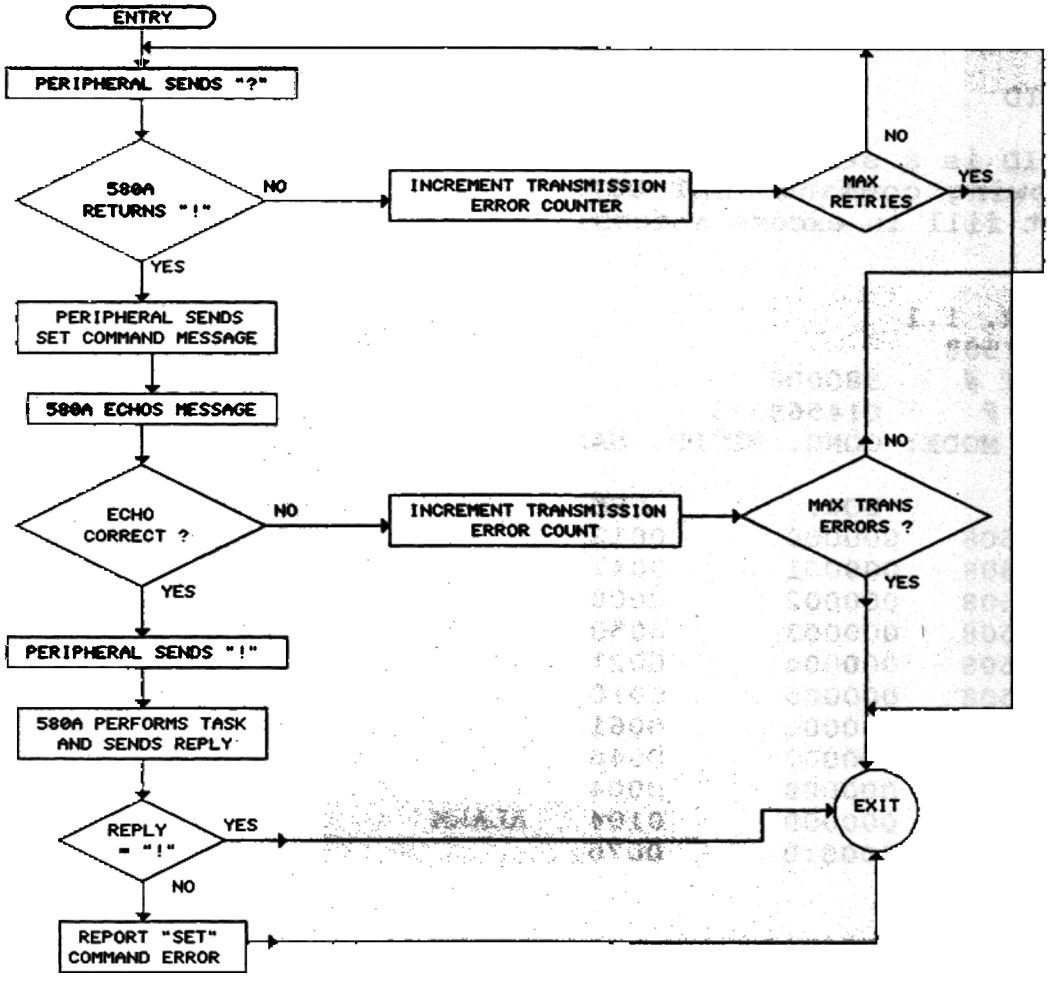


Figure A.4
Software Handshake
Set Commands

SET REAL TIME

The format for setting the real time is as follows:

```
II/II/II IIII
 .  .  .  . .
    .  .  . .........minutes (max=59)
    .  .  ..........hours (max=23)
    .  .............year
    ................day
    ................month
```

For example: SET REAL TIME 02/15/86 1723 (ret)
This instructs the 580B to set its real time clock to February 15, 1986 5:23 p.m.

SET USER ID

The user ID is a string of 9 integers. To set user ID = 66, use the following command "SET USER ID 000000066 (ret)" (leading zeros must fill in excess spaces).

```
580B   VER. 1.1
07/11/88 1508
INSTRUMENT #     580000
USER I.D. #      014569373
OPERATING MODE: CONC. METER, MAX HOLD

                  LOC.        PPM    STATUS
07/11/88 1508   000000       0012
07/11/88 1508   000001       0047
07/11/88 1508   000002       0000
07/11/88 1508   000003       0050
07/11/88 1508   000004       0021
07/11/88 1508   000005       0010
07/11/88 1509   000006       0061
07/11/88 1509   000007       0046
07/11/88 1509   000008       0004
07/11/88 1509   000009       0104   ALARM
07/11/88 1509   000010       0076

07/11/88 1509
INSTRUMENT #     580000
USER I.D. #      014569373
OPERATING MODE: CONC. METER

                  LOC.        PPM    STATUS
07/11/88 1509   000011       0000
07/11/88 1509   000012       0064
07/11/88 1509   000013       0052
07/11/88 1509   000014       0001
07/11/88 1509   000015       0007
07/11/88 1509   000016       0101   ALARM
```

Figure A.5
Data Log Output

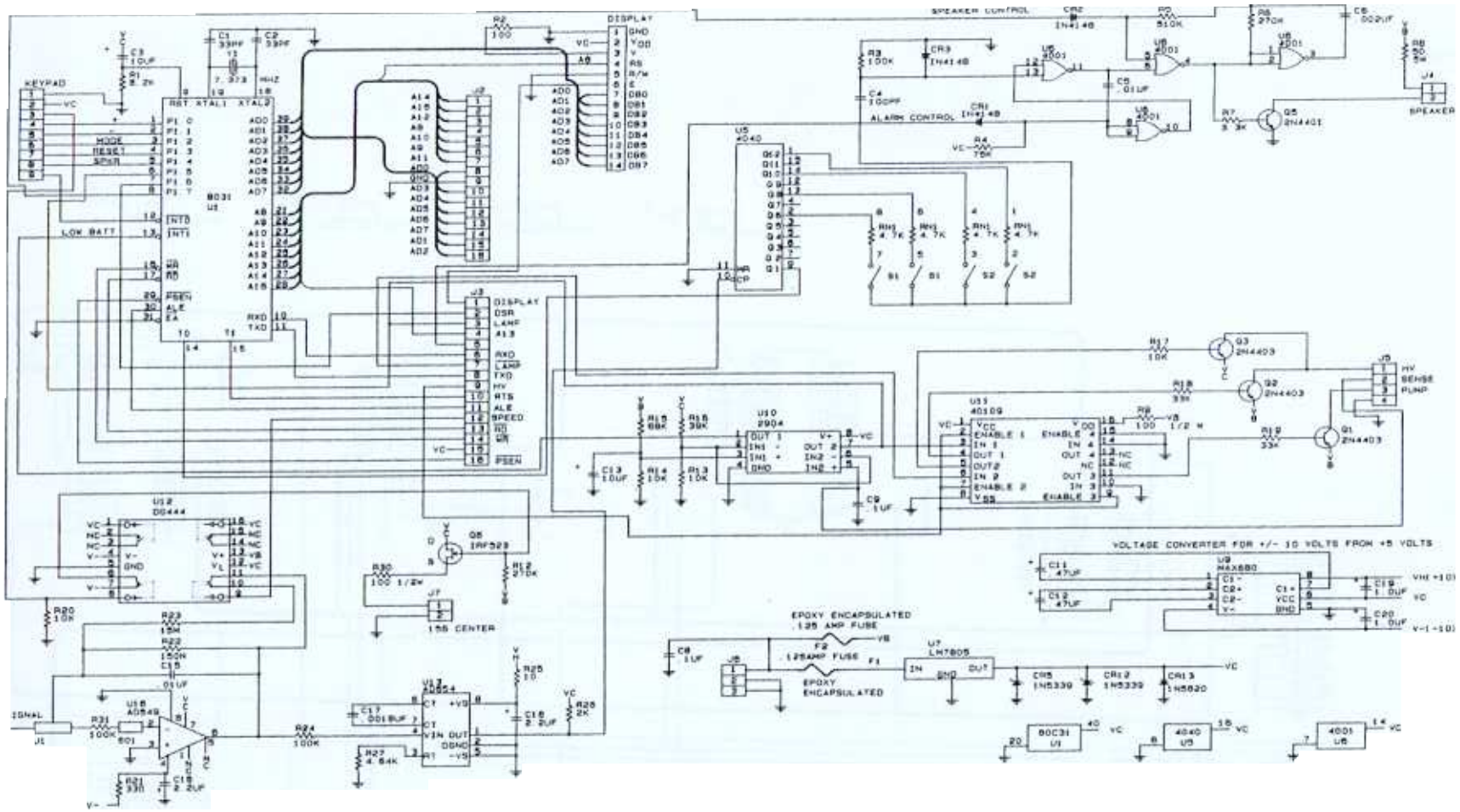


Figure B-1

Processor/Ranging Bd. Schematic

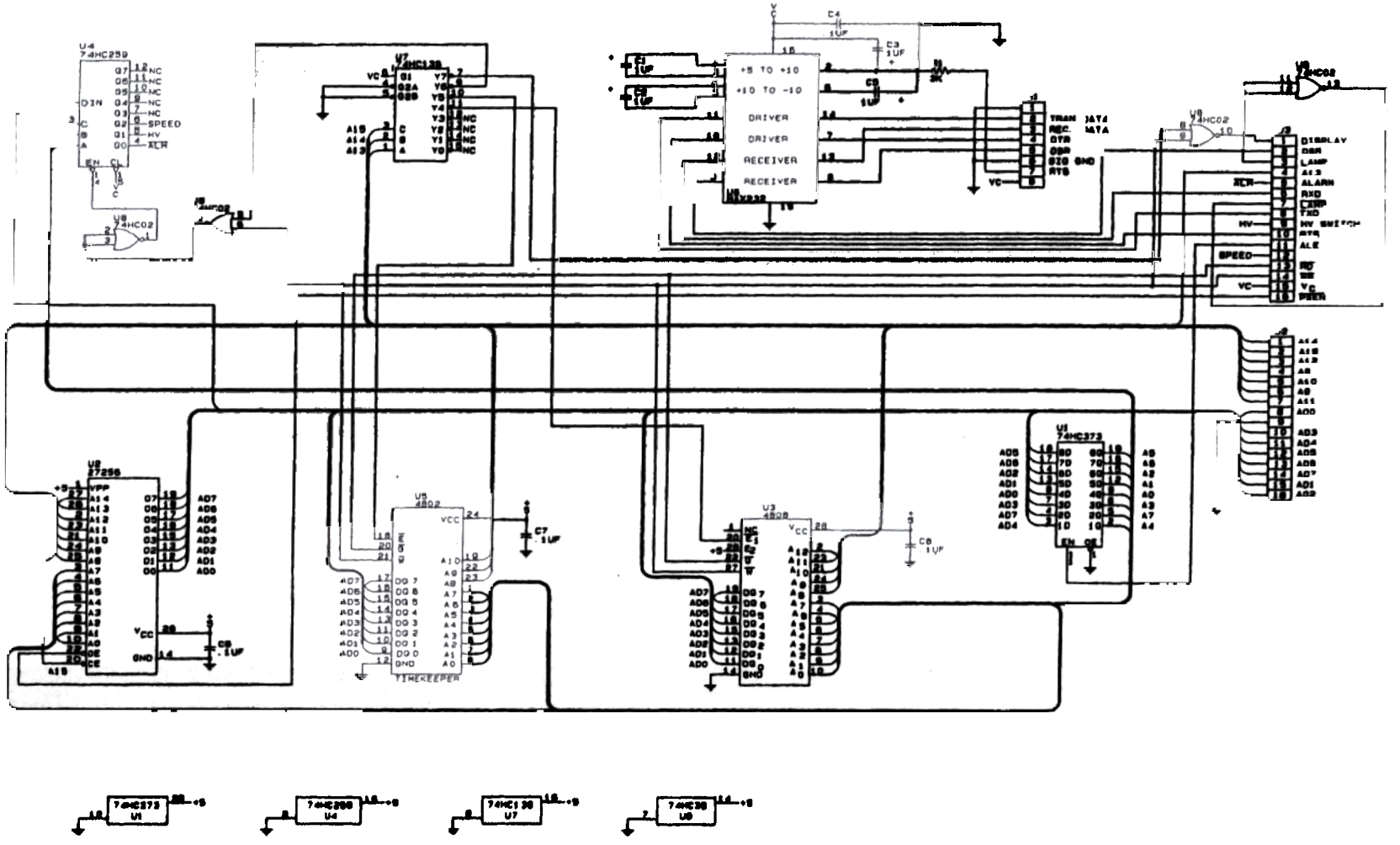
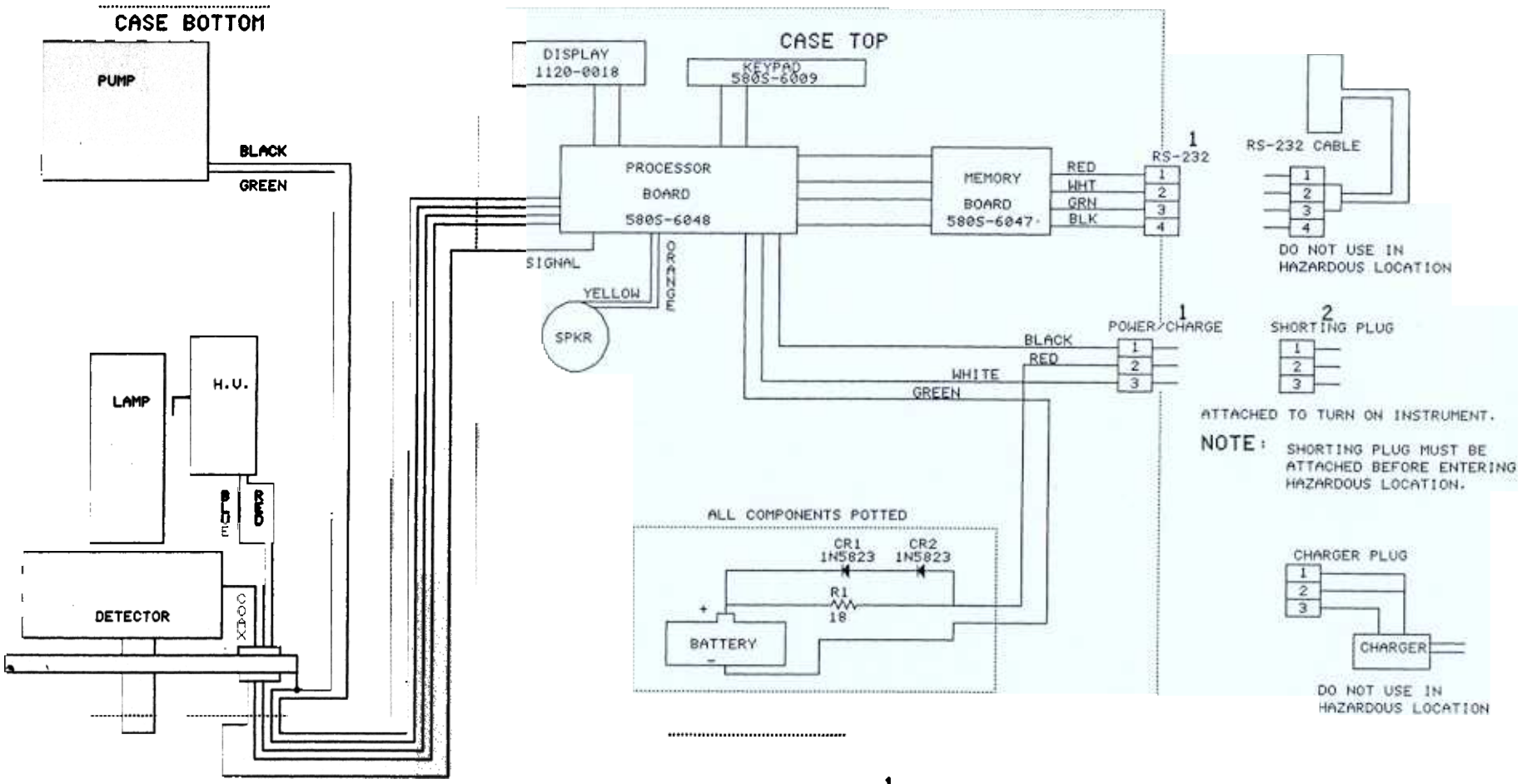


Figure B.2

Memory Board Schematic



1 CONNECTORS MOUNTED ON REAR OF CASE TOP.

2 SHORTING PLUG IS TETHERED TO PREVENT ITS LOSS.

Figure C.1
Block Diagram

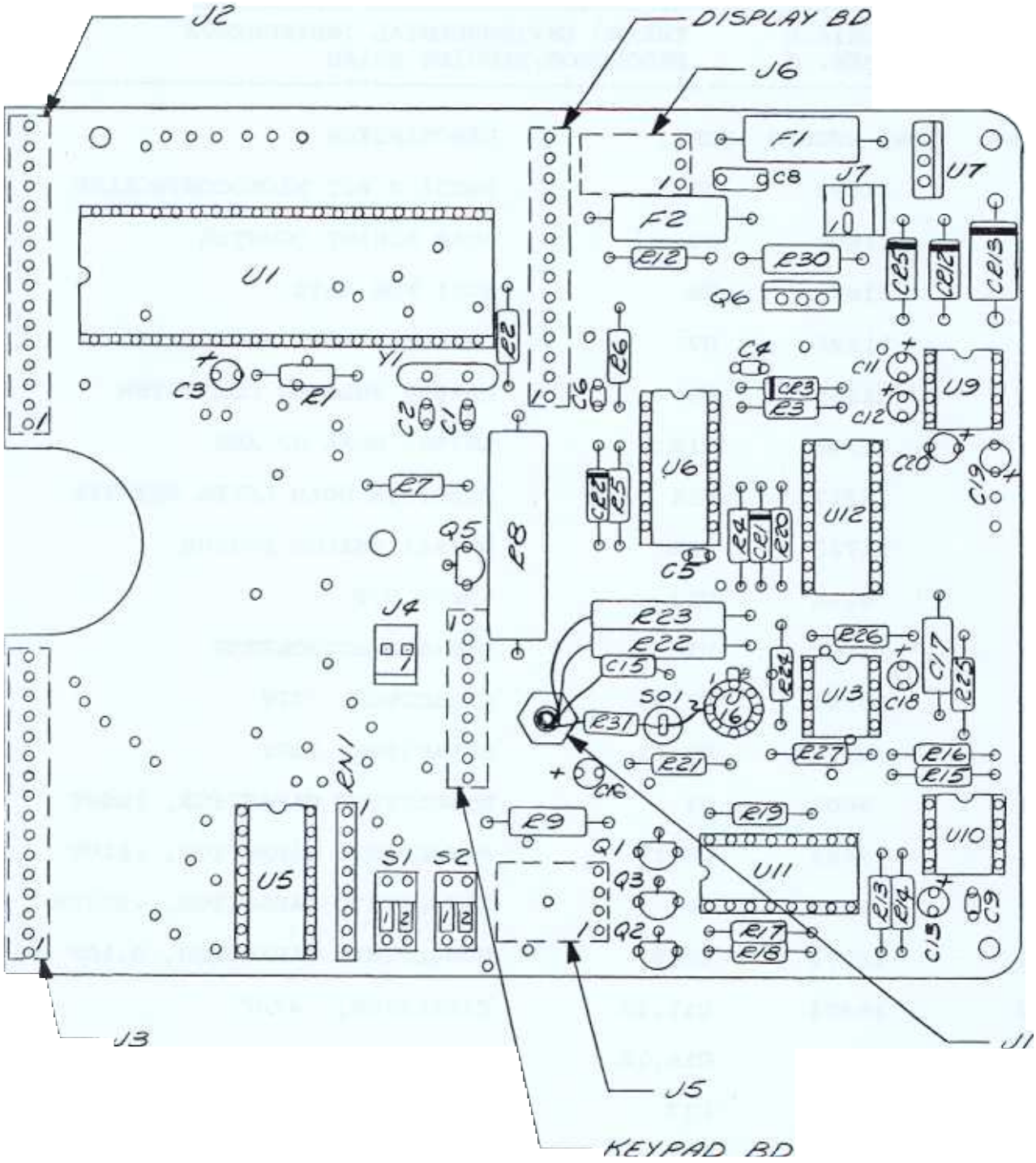


Figure D-1
Processor/Ranging Bd. Loading Drawing

BILL OF MATERIALS THERMO ENVIRONMENTAL INSTRUMENTS
580S-6048 REV. A PROCESSOR/RANGING BOARD

QUAN.	PART NUMBER	DESG.	DESCRIPTION
1	11695	U1	80C31 8 BIT MICROCONTROLLER
1	11696	U5	4040 BINARY COUNTER
1	11697	U6	4001 NOR GATE
1	11126	U7	LM7805 5 VOLT REGULATOR
1	11159	U9	MAX680 VOLTAGE CONVERTER
1	11716	U10	LM2904 DUAL OP AMP
1	11717	U11	CD40109B QUAD LEVEL SHIFTER
1	11723	U12	DG-444 ANALOG SWITCH
1	9296	U13	AD654 V/F
1	11152	U16	AD549 ELECTROMETER
2	10391	C1,2	CAPACITOR, 33PF
2	10376	C3,13	CAPACITOR, 10UF
1	5609	C4	MONOLITHIC CAPACITOR, 100PF
2	5605	C5,15	MONOLITHIC CAPACITOR, .01UF
1	10399	C6	MONOLITHIC CAPACITOR, .002UF
2	10372	C8,9	MONOLITHIC CAPACITOR, 0.1UF
2	10403	C11,12	CAPACITOR, .47UF
2	10390	C16,18	CAPACITOR, 2.2UF
1	10402	C17	POLYPROPYLENE CAP 1.8NF,5%
2	10378	C19,20	CAPACITOR, 1.0UF
1	10790	R1	RESISTOR, 8.2K
1	10847	R2	RESISTOR, 100, 1/4 W
3	10862	R3,24,31	RESISTOR, 100K
1	10928	R4	RESISTOR, 75K

BILL OF MATERIALS
580S-6048 REV. A

THERMO ENVIRONMENTAL INSTRUMENTS
PROCESSOR/RANGING BOARD

QUAN.	PART NUMBER	DESG.	DESCRIPTION
1	10929	R5	RESISTOR, 510K
2	10930	R6,12	RESISTOR, 270K
1	5986	R7	RESISTOR, 3,3K
1	10939	R8	RESISTOR, 50, 5W
2	10704	R9,30	RESISTOR, 100, 1/2W
4	10864	R13,14,17,20	RESISTOR, 10K
1	10938	R15	RESISTOR, 68K
1	6025	R16	RESISTOR, 39K
2	2219	R18,19	RESISTOR, 33K
1	10786	R21	RESISTOR, 330
1	10951	R22	RESISTOR, 150M, 5%
1	10952	R23	RESISTOR, 15M, 5%
1	10846	R25	RESISTOR, 10
1	10936	R26	RESISTOR, 2K
1	10865	R27	RESISTOR, 4.64K
1	11641	RN1	NETWORK, 4.7K X 6
3	11807	CR1-3	1N4148
2	11829	CR5,12	1N5339
1	11805	CR13	1N5820
1	10557	Y1	7.373 MHZ
1	10446	J1	RADIAL SMC
1	11398	J4	2 PIN AMP, .100 CENTER
1	11418	J5	4 PIN MTE

BILL OF MATERIALS THERMO ENVIRONMENTAL INSTRUMENTS
580S-6048 REV. A PROCESSOR/RANGING BOARD

QUAN.	PART NUMBER	DESG.	DESCRIPTION
1	11419	J6	3 PIN MTE
1	11405	J7	2 PIN AMP .156 CENTER
2	580S-6040	F1,2	.125 AMP FUSE ASSEMBLY
1	10422	S01	STANDOFF
2	12138	S1,2	SPST SWITCH, DUAL
3	11762	Q1-3	2N4403
1	11759	Q5	2N4401
1	11773	Q6	IRF523
1	580S-2057 REV. A		P.C. BOARD BLANK

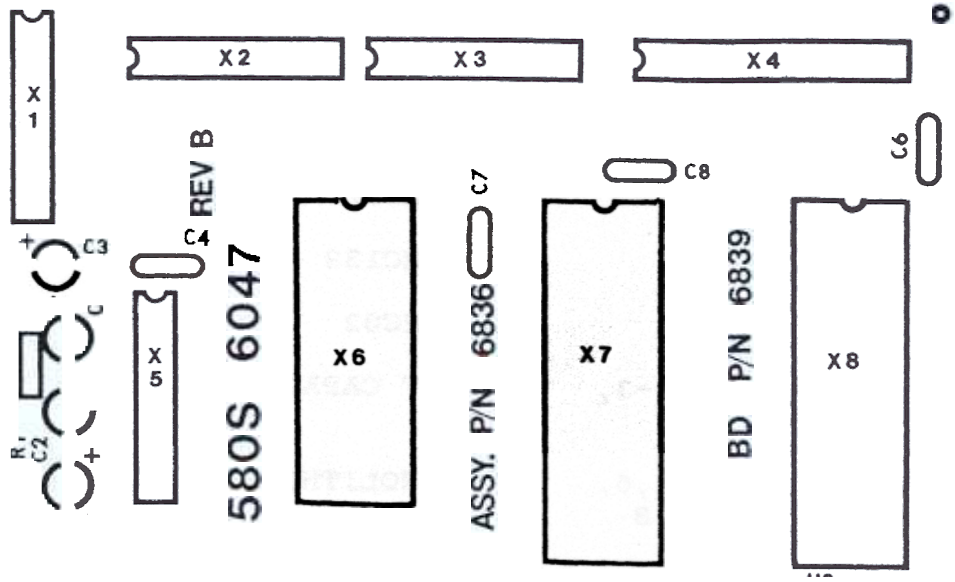
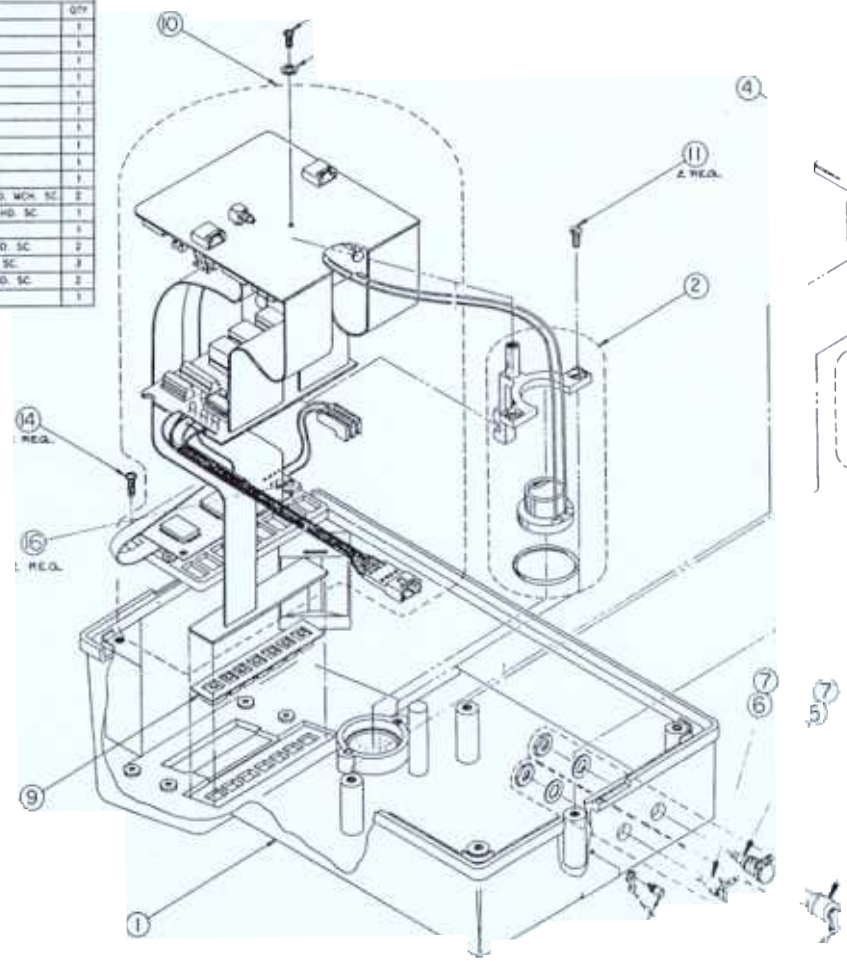


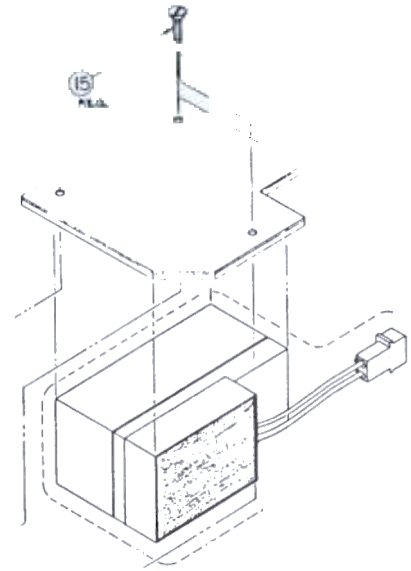
Figure D.2
Memory Board Loading Drawing

BILL OF MATERIALS THERMO ENVIRONMENTAL INSTRUMENTS
580S-6047 REV. A MEMORY BOARD

QUAN.	PART NUMBER	DESG.	DESCRIPTION
1	11689	U1	74HC373
1	11728	U2	27C256 8K EPROM
1	11687	U3	48Z08 8K X 8 BATTERY BACKED RAM
1	11688	U4	74HC259 OUTPUT PORT
1	11691	U5	48T02 2K X 8 TIMEKEEPER RAM
1	11685	U6	MAX232 RS-232 CHIP
1	11151	U7	74HC138
1	11730	U8	74HC02
4	10378	C1-3, 5	1UF CAPACITOR
4	10372	C4,6, 7,8	MONOLITHIC CAPACITOR, .1UF
1	10789	R1	RESISTOR, 3K
1	11374	X7	28 PIN SOCKET
1	580S-2056 REV. A		P.C. BOARD BLANK

MATERIAL LIST			
ITEM	PART NO.	DESCRIPTION	QTY
1	16801	CASE TOP	1
2	13675	SPEAKER ASSY.	1
3	13592	BATTERY ASSY.	1
4	13497	BATTERY HOLDER	1
5	13555	POWER HARNESS	1
6	5808-8029	RS-232 HARNESS ASSY.	1
7	11431	DUST COVER	1
8	16808	SHORTING PLUG	1
9	10460	KEYPAD	1
10	18538	P.C. BD SET UA	1
11	5868	8-32 X 1/4" LG. FLT. HD. MCH. SC	2
12	5815	4-40 X 5/16" LG. BND. HD. SC.	1
13	7534	NYLON WASHER	1
14	5856	8-32 X 1/2" LG. PAN. HD. SC.	2
15	12088	8-32 X 1" LG. FLAT HD. SC.	3
16	5822	8-32 X 5/8" LG. BND HD. SC.	2
17	6383	4-40 INSERT	1





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APPENDIX F
COMMON ORGANIC SOLVENTS AND GASES DATA SHEET

CHEMICAL MATERIALS	F.W. (G/MOLE)	DENSITY (G/ML)	BP (°C)*	I.P. (EV)**	TWA (PPM)***
Acetaldehyde	44.05	0.788	21	10.21	
Acetamide	59.07	1.159	221	9.77	
Acetic Acid	60.05	1.049	116-117	10.37	10
Acetic Anhydride	102.1	1.10	138-117	9.88	5
Acetone	58.1	0.79	56	9.69	1000
Acetonitrile	41.1	0.79	82	12.22	40 cell
Acetophenone	120.15	1.033	202	9.27	
Acetyl Bromide	122.96	1.52	75-75	10.55	
Acetyl Chloride	78.50	1.104	52	11.02	
Acetylene	26.02	0.90		11.41	
Acrolein	56.06	0.8389	53	10.10	0.1
Acrylonitrile	53.06	0.8004	77	10.91	10
Allyl Alcohol	58.1	0.85	96-98	9.67	2
Allyl Chloride	76.5	0.94	44-46	9.9	1
Aniline	93.1	1.02	184	7.70	5
Anisole	108.13	0.9956	154	8.22	
Ammonia	17.03	gas		10.15	
Arsine	77.9	gas			
Benzaldelyde	106.12	1.053	178-185	9.53	
Benzene	78.1	0.88	80	9.25	1
Benzonitrile	103.12	1.010	188	9.71	
Benzotriflouride	146.11	1.1886	102	9.68	
Benzyl Chloride	126.6	1.10	177-181	9.14	1
Biphenyl	154.21	0.992	255		
Bromine	159.81	3.1023	58.8	10.55	
Bromobenzene	157.02	1.495	156	8.98	
1-Bromobutene	137.03	1.276	100-04	10.13	
2-Bromobutene	137.03	1.255	91	9.98	
1-Bromo-2-Chlorethene	143.42	1.723	106-07	10.63	
Bromochloromethane	129.39	1.991	68	10.77	
1-Bromo-2-Flourobenzene	175.01	1.593	150	8.99	
Bromoform	252.8	2.9	150-01	10.47	0.5
1-Bromo-2-methyl propane	137.03	1.260	90-92	10.09	
2-Bromo-2-methyl propane	137.03	1.189	72-74	9.89	
1-Bromopentane	151.05	1.218	130	10.10	
2-Bromopropane	123.00	1.354	71	10.18	
2-Bromopropene	123.00	1.310	59	10.08	
1-Bromopropene	120.98	1.413	58-63	9.30	
3-Bromopropene	120.98	1.398	70-71	9.70	
2-Bromothiophene	163.04	1.684	149-151	8.63	
M-Bromotoluene	171.04	1.4099	183.7	8.81	
O-Bromotoluene	171.04	1.431	58.60	8.79	
P-Bromotoluene	171.04	1.431	184	8.67	
1,3-Butadiene	54.1	gas	9.07		1000
Butane	58.12	gas	10.63		
	90.19	.842	9.14		

* BP - Boiling Point Degrees Centigrade
** IP - Ionization Potential
*** TWA - Time Weighted Average = Parts Per Million

CHEMICAL MATERIALS	F.W. (G/MOLE)	DENSITY (G/ML)	BP (°C)*	I.P. (EV)**	TWA (PPM)***
2-Butanone	72.1	0.81	80	9.53	200
1-Butene	56.10	0.6255		9.58	
N-Butyl Acetate	116.2	0.88	124-26	10.01	150
S-Butyl Acetate	116.2	0.88	111-12	9.91	150
N-Butyl Alcohol	74.1	0.81	117.7	10.04	100
N-Butyl Amine	73.1	0.73	73	78	5
S-Butyl Amine	73.1	0.73	63	8.70	5
T-Butyl Amine	73.1	0.73	46	8.64	5
N-Butyl Benzene	134.21	0.8604	183	8.69	
S-Butyl Benzene	134.21	0.8604	173-04	8.68	
T-Butyl Benzene	134.21	0.8669	169	8.68	
N-Butyraldehyde	72.10	0.8016	75	9.86	
N-Butyric Acid	88.10	0.959	162	10.16	
N-Butyronitrile	69.10	0.7954	115-17	11.67	
Camphor	152.2	0.99	204	8.76	2 mg/m
Carbon Dioxide	44.01	gas		13.79	5000
Carbon Monoxide	28.01	gas		14.01	50
Carbon Tetrachloride	153.8	1.59	77	11.47	10
Chlorine	70.90	gas		11.48	1 cell
Chlorobenzene	112.6	1.10	132	9.07	75
Chloroform	119.4	1.48	60.5-61.5	11.37	50 cell
1-Chloro-2-Methylpropane	92.57	.883	68-69	10.66	
2-Chloro-2-Methylpropane	101.64	.851	51-52	10.61	
1-Chloropropane	78.54	.892	46-47	10.82	
2-Chloropropane	78.54	.859	34-36	10.78	
3-Chloropropane	76.53	.939	44-46	10.04	
2-Chlorothiophene	118.59	1.286	127-29	8.68	
M-Chlorotoluene	126.58	1.076	160-162	8.83	
O-Chlorotoluene	126.58	1.0826	157-159	8.83	
P-Chlorotoluene	126.58	1.0697	162	8.70	
M-Cresol	108.1	1.034	203	8.52	5 cell
O-Cresol	108.1	1.048	191	8.50	5 cell
P-Cresol	108.1	1.034	202	8.38	5 cell
Crotonaldehyde	70.09	0.853	104	9.73	2
Cumene	120.2	0.86	152-154	8.75	50
Cyanogen	52.04	0.9537		13.80	
Cyclohexane	84.2	0.81	80.7-81	9.98	300
Cyclohexane	100.2	0.96	160-161	10.0	50
Cyclohexanone	98.1	0.95	155	9.14	50
Cyclohexene	82.1	0.81	83	8.95	300
Cyclo-Octatetraene	104.15	0.925	142-43	7.99	
Cyclopentane	70.13	0.7460	50	10.53	
Cyclopentanone	84.11	1.4366	130-131	9.26	
Cyclopentene	68.12	0.744	44	9.01	
Cyclopropane	42.08	gas		9.91	
Diborane	27.68	gas		11.00	
Diazomethane	42.0	gas		9.0	0.2

* BP - Boiling Point Degrees Centigrade
** IP - Ionization Potential
*** TWA - Time Weighted Average = Parts Per Million

CHEMICAL MATERIALS	F.W. (G/MOLE)	DENSITY (G/ML)	BP (°C)*	I.P. (EV)**	TWA (PPM)**
Dibromodiflouromethane	209.83	2.297	22-23	11.07	
1,2-Dibromoethane	187.87	2.180	131-32	9.45	
1,3-Dibromopropane	201.90	1.937	167	10.07	
Dibutylphthlate	278.3	1.04	340		5 mg/m
M-Dichlorobenzene	147.01	1.288	172-73	9.12	50
O-Dichlorobenzene	147.01	1.306	179-180	9.07	50
P-Dichlorobenzene	147.01	1.241	173	8.94	75
1,1-Dichlorethane	99.0	1.18	57	11.06	100
1,2-Dichlorethane	98.96	1.256	83	11.12	
1,2-Dichlorethylene	97.0	1.28	46-60	9.66	
Dichloromethane	84.93	1.325	39.8-40	11.35	
1,2-Dichloropropane	112.99	1.156	95-96	10.87	
1,3-Dichloropropane	112.99	1.190	120-22	10.85	
2,3-Dichloropropane	110.97	1.204	94	9.82	
N,N-Diethyl Acetamide	115.18	0.925	182-86	8.60	
Diethylamine	73.1	0.71	55	8.01	25
Diethyl Ether	74.12	0.7134	34.6	9.53	
N.N-Diethyl Formamide	101.15	0.908	176-77	8.89	
Diethyl Ketone	86.13	0.816	102	9.32	
Diethyl Sulfide	90.19	.837		8.43	
Diethyl Sulfite	138.19	1.883	158-60	9.68	
Dihydropyran	84.12	0.922	86	8.34	
Diisopropylamine	101.2	0.72	84	7.73	5
1,1-Dimethoxyethane	90.12	0.863	64	9.65	
N,N-Dimethyl Acetamide	87.12	0.937	164.5-66	8.81	10
Dimethyl Amine	45.1	0.68		8.24	10
N,N-Dimethyl Aniline	122.2	0.96	193-94	7.13	
2,2-Dimethyl Butane	86.18	0.649	50	10.06	
2,3-Dimethyl Butane	86.18	0.662	50	10.02	
3,3-Dimethyl Butanone	100.16	0.801	106	9.17	
N,N-Dimethyl Formamide	73.09	0.9445	153	9.12	10
Dimethlyl Sulfide	63.13	0.846	38	8.69	
P-Dioxane	88.1	1.03	100-102	9.13	
Dipropyl Amine	101.19	0.738	105-110	7.84	
Durene	134.12	0.84	80-82	8.03	
Epichlorohydrin	92.5	1.18	115-117		5
Ethane	30.07	gas		11.65	
Ethanethiol	62.13	0.8315	35	9.29	
Ethyl Acetate	88.1	0.90	76.5-77.5	10.11	400
Ethyl Alcohol	46.1	0.80	78	10.48	1000
Ethyl Amine	45.1	0.69	19.20	8.86	10
Ethyl Benzene	106.2	0.87	136	8.76	100
Ethyl Bromide	109.0	1.45	37-40	10.29	200
Ethyl Butyl Ketone	114.2	0.82	146-49	9.02	50
Ethyl Chloride	64.52	0.9214		10.98	1000
Ethyl Disulfide	122.25	0.993	153	8.27	
Ethylene Dibromide	187.9	2.17	131-132	10.52	20
Ethylene Dichloride	99.0	1.26	83	11.32	50

* BP - Boiling Point Degrees Centigrade
** IP - Ionization Potential
*** TWA - Time Weighted Average = Parts Per Million

CHEMICAL MATERIALS	F.W. (G/MOLE)	DENSITY (G/ML)	BP (°C)*	I.P. (EV)**	TWA (PPM)**
Ethyl Ether	74.1	0.73	34.6	9.59	400
Ethyl Formate	74.1	0.92	52-54	10.61	100
Ethyl Iodide	155.98	1.950	67-73	9.33	
Ethyl Isothiocyanate	87.15	1.003	60	9.14	
Ethyl Methyl Sulfide	76.16	0.842	66-67	8.55	
Ethyl Nitrate	75.07	0.90	112	11.22	
Ethyl Propionate	102.13	0.891	99	10.00	
Ethyl Thiocyanate	87.14	1.007		9.89	
Ethynylbenzene	102.13	0.9300	142-44	8.82	
Fluorine	37.99	gas		15.70	
Flourobenzene	96.10	1.024	85	9.20	
O-Fluorophenol	112.10	1.256	172-74	8.95	
M-Fluorotoluene	110.13	0.997	178	8.92	
O-Fluorotoluene	110.13	1.004	172-172	8.92	
P-Fluorotoluene	110.13	1.001	185	8.79	
Formaldehyde	30.03	1.083		10.87	3
Formahide	45.04	1.1334	210	10.25	
Formic Acid	46.02	1.220	110-101	11.05	
2-Furaldehyde	96.09	1.160	182	9.21	
Furan	68.07	0.9371		8.89	
Heptane	100.2	0.68	98	10.08	
2-Heptanone	114.18	0.8068	149-50	9.33	
Hexane	86.2	0.66	68-69	10.18	500
1-Hexane	84.16	0.673	64	9.46	
Hexone	100.2	0.80		9.53	
Hydrogen	2.017	gas		15.43	
Hydrogen Bromide	80.92	gas		11.62	3
Hydrogen Chloride	36.47	gas		12.74	5 cell
Hydrogen Cyanide	27.03	0.687		13.91	10
Hydrogen Flouride	20.01	gas		15.77	3
Hydrogen Iodide	127.93	gas		10.38	
Hydrogen Selenide	80.98	gas		9.88	0.05
Hydrogen Sulfide	34.08	gas		10.46	20 cell
Hydrogen Telluride	129.63	gas		9.14	
Iodine	253.81	4.93		9.28	0.1 cell
Iodobenzene	204.02	1.8384	188	8.73	
1-Iodobutene	184.02	1.617	130-31	9.21	
2-Iodobutene	184.02	1.4991	119-120	9.09	
1-Iodo-2-Methylpropane	184.02	1.599	120-21	9.18	
1-Iodopentane	198.05	1.517	154-55	9.19	
1-Iodopropane	169.99	1.743	101-02	9.26	
2-Iodopropane	169.99	1.703	88-90	9.17	
O-Iodotoluene	218.04	1.713	211	8.62	
M-Iodotoluene	218.04	1.698		8.61	
P-Odotoluene	218.04		211-5	8.50	
Isoamyl Acetate	130.2	0.88	142	9.94	
Isoamyl Alcohol	88.2	0.81	130-1	10.42	

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CHEMICAL MATERIALS	F.W. (G/MOLE)	DENSITY (G/ML)	BP (^{o}C)*	I.P. (EV)**	TWA (PPM)**
Isobutyl Amine	73.14	0.724	64-71	8.70	
Isobutyl Formate	102.13	0.885	98.4	10.46	
Isobutylene	56.11	0.5942	-6.9	9.23	
Isobutyraldehyde	72.11	0.794	63	9.74	
Isobutyric Acid	88.11	0.950	153-54	10.02	
Isoctane	114.2	0.70	98-99	17.9	
Isopentane	114.23	0.692	30	10.32	
Isoprene	68.12	0.681	34	8.85	
Isopropyl Acetate	102.1	0.87	85	9.99	250
Isopropyl Alcohol	60.1	0.79		10.16	400
Isopropyl Amine	59.1	0.69	33-34	8.72	5
Isopropyl Benzene	120.2	0.86	152-54	8.75	50
Isopropyl Ether	102.2	1.37	68-69	9.20	500
Isovaleraldehyde	86.13	0.785	90	9.71	
2,3-Lutidine	107.15	0.945	162-63	8.85	
2,4-Lutidine	107.15	0.927	159	8.85	
2,6-Lutidine	107.15	0.9252	143-45	8.85	
Malaic Anhydride	98.1	0.93	200	11.1	
Mesitylene	120.19	0.8637	162-64	8.40	
Mesityl Oxide	98.14	0.8592	129	9.08	
Methane	16.04	gas		12.98	
Methanethiol	48.11	0.96		9.44	
N-Methyl Acetamide	73.10	0.957	204-05	8.90	
Methyl Acetate	74.08	0.9279	57.5	10.27	
Methyl Acrylate	86.1	0.96	80	9.9	10
Methyl Amine	31.06	gas	48	8.97	
Methyl Bromide	95.0	gas		10.53	
2-Methyl-1-Butane	70.16	0.650	31	9.12	
3-Methyl-1-Butane	70.14	0.627	20	9.51	
3-Methyl-2-Butane	70.14	0.643		8.67	
Methyl Butyl Ketone	100.6	0.83	127	9.34	
Methyl Butyrate	102.13	0.898	102-103	10.07	
Methyl Chloride	50.5			11.28	
Methyl Cyclohexane	98.19	0.770	101	9.85	
Methyl Disulfide	94.20	1.046	109	8.46	
Methyl Ethyl Ketone	72.10	0.805	80	9.53	
Methyl Formate	60.1	1.34	34	10.815	100
2-Methyl Furan	82.10	0.827	63-66	8.39	
Methyl Iodide	142.0	2.28	41-43	9.54	5
Methyl Isobutyl Ketone	100.2	0.80	117-18	9.30	100
Methyl Isobutyrate	102.13	0.891	90	9.98	
Methyl Isopropyl Ketone	86.12	0.805	94-95	9.32	
Methyl Isothiocyanate	73.12		37-39	9.25	
Methyl Methacrylate	100.1	0.94	100	9.9	100
1-Methyl Napthalene	142.20	1.001	240-243	7.96	
2-Methyl Napthalene	142.20	1,000	241-242	7.96	
2-Methyl Pentane	86.18	0.653	62	10.12	
3-Methyl Pentane	86.18	0.664	64	10.08	
Methyl Propionate	88.11	0.915	79	10.15	

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CHEMICAL MATERIALS	F.W. (G/MOLE)	DENSITY (G/ML)	BP (°C)*	I.P. (EV)**	TWA (PPM)**
Methyl Propyl Ketone	86.13	0.809	100.01	9.38	
2-Methyl Styrene	165.4	1.068	131	10.07	
Monomethyl Aniline	107.16	0.989			
Monomethyl Hydrazine	46.1	0.87			
Morpholine	87.1	1.01	129	8.88	20
Nephthalene	93.7	1.16	217.7	8.12	10
Nitric Oxide	162.2	1.01		9.25	
P-Nitroaniline	138.1				1.0
Nitrobenzene	123.1	1.21	210-211	9.92	1.0
4-Nitrobiphenyl	199.2				
P-Nitrochlorobenzene	157.6	1.52		9.96	1.0 mg/m
Nitrogen Dioxide	46.01	1.448		9.78	
Nitroethane	75.1	1.38	112	10.81	100
Nitromethane	61.0	1.13	100.8-101	11.08	100
1-Nitropropane	89.1	0.99	131-32	10.88	25
2-Nitropropane	89.1	0.98	120	10.71	25
N-Nitrosodimethylamine	74.1	1.00	153	9.07	
Nitrotoluene	137.1	1.16	225-238	11.63	5
Oxygen	31.9988	gas		12.08	
Ozone	48.00	gas		12.08	
Pentaborane	63.17	0.61		10.40	
Pentane	72.15	0.62638	35	10.35	
2,4-Pentanedione	70.13	0.6429	140.4	8.87	
1-Pentene	70.13	0.6503	29.9-30.1	9.50	
Phenetol	122.16	0.967	169-70	8.13	
Phenol	94.1	1.07	182	8.50	5
Phenyl Hydrazine	108.1	1.1	238-41	7.86	5
Phenyl Isocyanate	119.12	1.0887	162-63	8.77	
Phenyl Isothiocyanate	135.18	1.1288	221	8.52	
Phosgene	98.9	gas		11.77	0.4 mg/m
Phosphine	34.0	gas			0.3
Phosphorous Pentachloride	208.2	1.6		10.7	1 mg/m
Phosphorous Trichloride	137.3	1.57	76	10.5	
2-Picoline	93.12	0.950	128-29	9.02	
3-Picoline	93.12	0.9613	144	9.02	
4-Picoline	93.12	0.9571	145	9.04	
Propane	44.09	gas		11.07	
1-Propanethiol	76.16	0.841	67-68	9.20	
Propiolactone	72.06	1.146	162	9.70	
Propionic Acid	74.08	0.99336	141	10.24	
Propionaldehyde	58.08	0.8071	46-50	9.98	
Propionitrile	55.08	0.7818	97	11.84	
N-Propyl Acetate	102.1	0.84	120	10.04	200
Propyl Alcohol	60.10	0.804	97	10.20	
Propyl Amine	59.11	0.719	48	8.78	
Propyl Benzene	120.20	0.862	159	8.72	
Propylene	42.08	gas		8.73	
Propylene Oxide	58.08	0.859	34	10.22	

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CHEMICAL MATERIALS	F.W. (G/MOLE)	DENSITY (G/ML)	BP (°C)*	I.P. (EV)**	TWA (PPM)***
Methyl Propyl Ketone	86.13	0.809	100.01	9.38	
2-Methyl Styrene	165.4	1.068	131	10.07	
Monomethyl Aniline	107.16	0.989			
Monomethyl Hydrazine	46.1	0.87			
Morpholine	87.1	1.01	129	8.88	20
Napthalene	93.7	1.16	217.7	8.12	10
Propyl Ether	102.17	0.7360	88.90	9.27	
Propyl Formate	88.10	0.901		10.54	
Pyrene	202.3	gas		7.41	
Pyridine	79.1	0.98	115	9.32	5
Pyrrole	67.09	0.9691	131	8.20	
Styrene	104.14	9.9059	145-146	8.47	
Styrene Oxide	120.2	1.054	194	9.04	
Tetrachloroethylene	165.9	1.63	121	9.32	100
Tetrahydrofuran	72.10	0.8892	67	9.54	
Tetrahydropyran	86.13	0.8814	88	9.26	
Thiophene	84.1	1.53	84	8.86	
Toluene	93.13	0.866	111	8.82	
O-Toluidine	107.2	1.01	199-200	7.44	5
Trichloroethene	131.40	1.4649	87	9.45	
Triethylamine	101.19	1.069	88.18	7.50	
Trimethyl Amine	59.11	0.636	3-4	7.82	
2,2,4-Trimethyl Pentane	114.23	0.692	98-99	9.86	
Tripropyl Amine	143.27	0.753	155-58	7.23	
Valeraldehyder	86.13	0.8095	103	9.82	
Valeric Acid	102.13	0.939	185	10.12	
Vinyl Acetate	118	0.94	72-73	9.19	10
Vinyl Bromide	106.96	1.517	16	9.80	
Vinyl Chloride	62.5	gas		10.00	1
Water	18.016	1.00	100	12.59	
M-Xylene	106.16	0.8684	138-39	8.56	100
O-Xylene	106.16	0.8801	143-45	8.56	100
P-Xylene	106.16	0.8614	138	8.45	100

* BP - Boiling Point Degrees Centigrade
** IP - Ionization Potential
*** TWA - Time Weighted Average = Parts Per Million

APPENDIX G

DILUTION PROBE OPERATION

(Not investigated as part of UL classified product)

G.1 GENERAL

The dilution probe is constructed of stainless steel and Teflon, with a charcoal filter mounted on the dilution inlet. The purpose of the charcoal filter is to provide hydrocarbon free air to the probe assembly so that the dilution of the incoming sample is not affected by the dilution air. The charcoal filter should be changed every 3 months to ensure proper operation, eliminating the problem of hydrocarbon breakthrough. It is easy to evaluate the performance of the charcoal, by challenging the Model 580A with hydrocarbon free air, then introducing a standard through the charcoal filter, with the inlet of the probe plugged. If there is breakthrough, a reading other than zero will be observed on the readout.

Another important part of the dilution probe is the 10 micron filter that is placed in the inlet of the probe assembly. The flow through this may reduce with time, as dirt collects on the inlet filter. This filter should be changed on a regular basis, depending upon the operator's experience and the environment in which he is working.

It is important to realize that the charcoal filter is not a totally efficient device. This does not cause a problem with the 580A, however, because the photoionization detector does not respond to ethane or methane.

TECHNICAL CONSIDERATION

Need For Dilution - The Model 580B dilution system was developed to increase the dynamic range of the Model 580A. As the instrument is manufactured, it has a workable range of 0 to 2,000 ppm. Above this upper limit, the detector is found to be non-linear. It does not absorb ethane or methane. The 580B will "lock out" for concentrations above 2,000 ppm. To meet the requirements of fugitive emission measurements as defined in EPA Method 21, there is the need to make measurements above the 2,000 ppm level. To accomplish this using a detector system that is limited by linearity, a dilution probe was developed. this probe provides a nominal 10 to 1 dilution ratio, increasing the dynamic range of the Model 580B from 2,000 to 20,000 ppm.

G.3 CALIBRATION OF THE DILUTION PROBE

The dilution probe is not factory calibrated. It has been tested and evaluated proper performance. It is the responsibility of the operator to properly calibrate the dilution probe.

The following is a simple procedure for this activity:

1. The performance of the 580B should have previously seen verified and calibrated.

2. Place the 580B in close proximity to a standard with the appropriate range. For example, if the instrument is to be used in a 5,000 ppm sampling range, then a standard of that concentration should be selected.

3. Connect the dilution probe making sure that the charcoal filter and the 10 micron filter are in place to the front of the 580B.

4. Challenge the instrument with the new standard gas and adjust the micro metering valve until a tenth of the reading is seen on the instrument readout.

5. As in the example of the 5,000 ppm standard, 500 ppm should be seen on the readout.

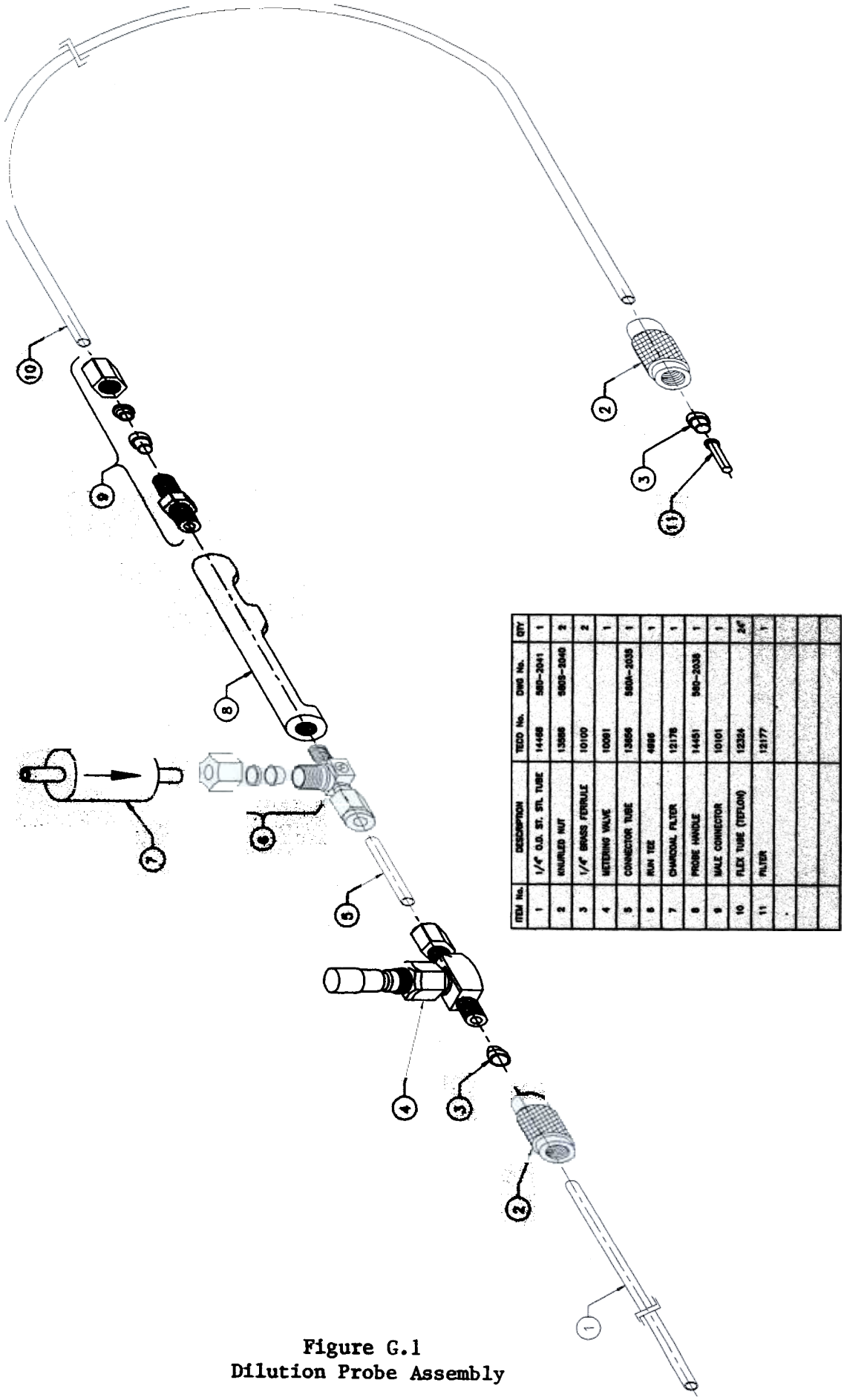
6. This is all that is required to calibrate the 580A or 580B.

   Note: It is important that both zero and span of of the 580A have been properly verified prior to initiating this procedure. It is very simple after using the dilution probe, to remove it and recheck the performance of the instrument on your low concentrator standard.

7. It should be noted that due to the environment that you are operating in, there may be a change in the back pressure of the charcoal filter and the 10 micron filter. Any changes in these over a period of time will cause a change in the split ratio of the dilution probe. Therefore, it is important to calibrate the dilution probe as regularly as you calibrate the 580A or 580B.

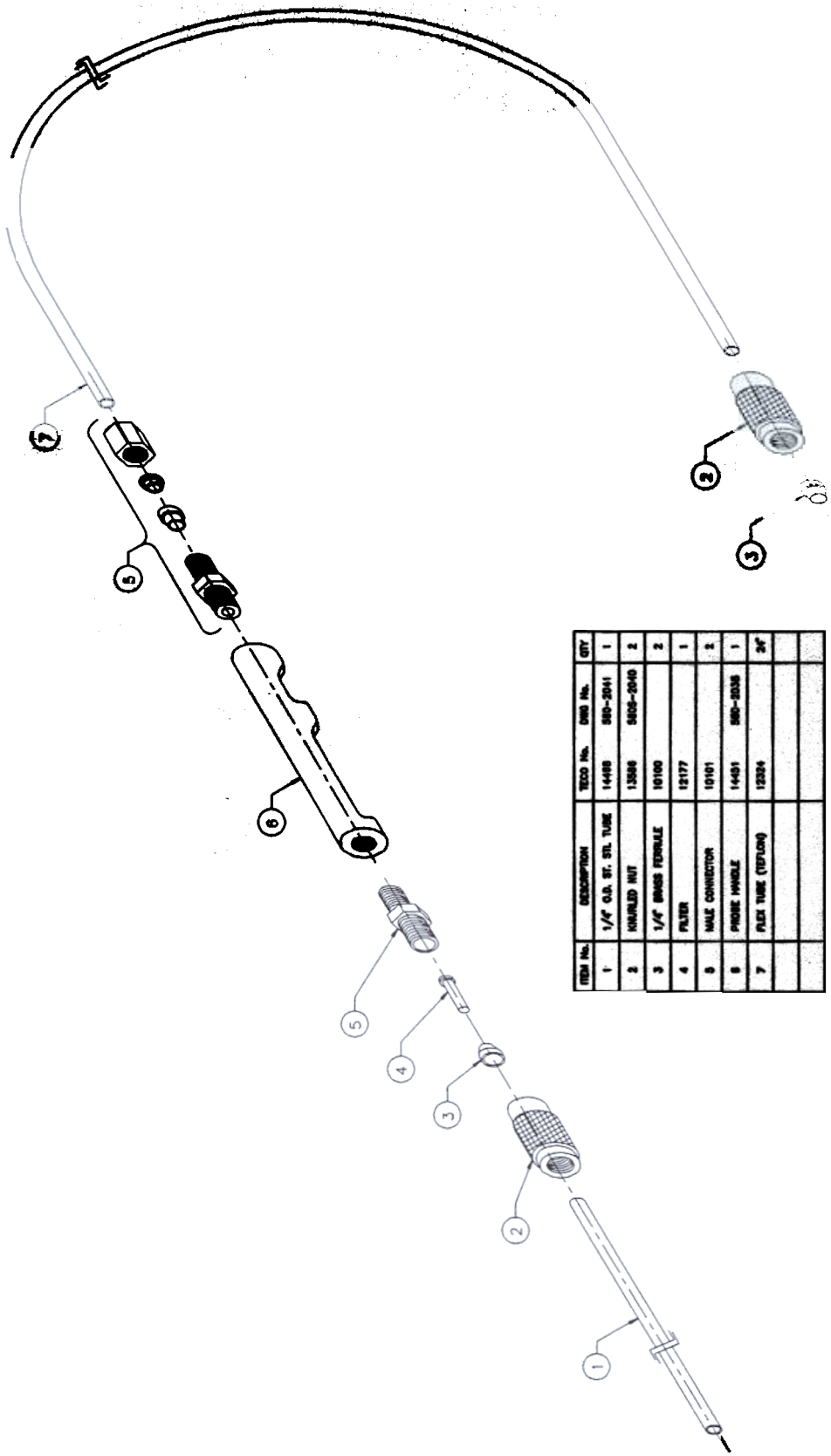
REPLACEMENT PARTS

1.	Inlet Probe Assembly	580A-6016
2.	Charcoal Filter	3150-0018
3.	Inlet Filter 10 Micron	3150-0017



ITEM No.	DESCRIPTION	TECO No.	DWG No.	QTY
1	1/4" O.D. ST. STL TUBE	14468	500-2041	1
2	KNURLED NUT	13569	5609-2040	2
3	1/4" BRASS FERRULE	10100		2
4	METERING VALVE	10091		1
5	CONNECTOR TUBE	13656	5604-2035	1
6	RUN TEE	4686		1
7	CHARCOAL FILTER	12178		1
8	PROBE HANDLE	14451	500-2039	1
9	MALE CONNECTOR	10101		1
10	FLEX TUBE (TEFLON)	12324		24'
11	FILTER	12177		1

Figure G.1
Dilution Probe Assembly



ITEM No.	DESCRIPTION	TECO No.	DWG No.	QTY
1	1/4" O.D. ST. STL TUBE	14499	SBD-2041	1
2	KNURLED NUT	13586	SBDS-2040	2
3	1/4" BRASS FERRULE	10100		2
4	FILTER	12177		1
5	MALE CONNECTOR	10101		2
6	PROBE HANDLE	14451	SBD-2036	1
7	FLEX TUBE (TEFLON)	12324		24"

Figure H.1
Probe Extension Assembly

APPENDIX I

INSTRUCTION SHEET
OPTION 11 WATER TRAP ASSEMBLY

(Not investigated as part of the UL classified product)

INSTALLATION

The water trap assembly (16846) is to be installed on the end of the 580 sample probe. The tygon tubing included is to be placed on the syringe side of the filter then connected to the probe. Refer to the drawing below.

USE

The water trap will effectively stop water from entering the instrument. Water traps can be re-used after drying at room temperature, but the user must be cautious of possible contamination. Contaminated traps should be discarded.

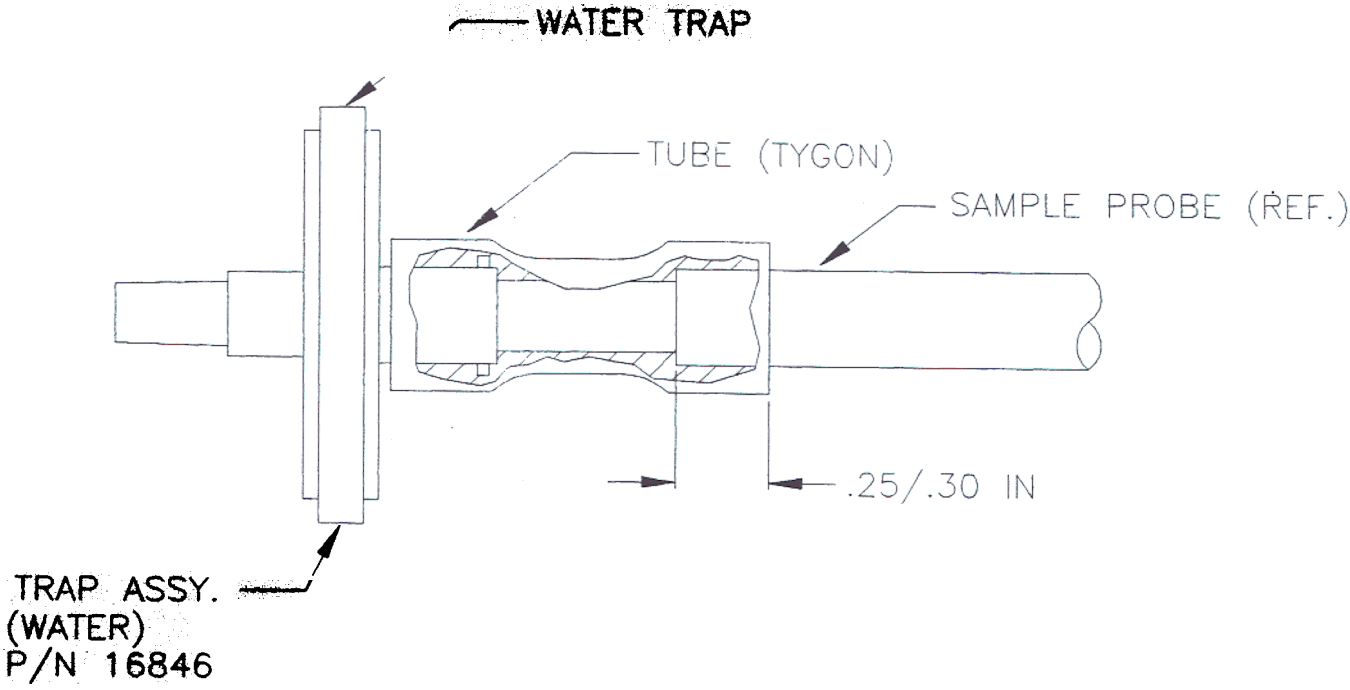


Figure I.1
Water Trap Assembly

APPENDIX J

Revised documents having an earlier revision level to the existing manual documents.

B-1	Processor Circuit	580S-9002	Sht. 1
B-2	Processor Circuit	580S-9002	Sht. 2
B-3	Memory Circuit	580S-9007	
C-1, 2, 3	Processor Board Bill of Materials	580S-6008	
	Processor Board Silkscreen	580S-6008	
	Memory Board Bill of Materials	580S-6007	
	Memory Board Silkscreen	580S-6007	

APPENDIX K

RS-232 Information Sheet

This procedure will retrieve logged data from Thermo Environmental Instruments' Model 580, and download the data into the HyperTerminal program for Windows 95 or Windows NT.

Hardware needed:

- Model 580
- Personal computer running either Win95 or WinNT with an available free serial communication port.
- RS-232 cable supplied with the Model 580

Software needed:

- HyperTerminal (supplied with Win95 and WinNT)

NOTE:
Even though this document uses HyperTerminal as the communication package, it is not necessary to use this program. HyperTerminal was chosen because it is part of the Win95 and WinNT software packages and is therefore readily available to users of these operating systems. Any communication program can be used to retrieve data from the Model 580 as long as it allows direct connections to a free communication port.

Procedure for setting up HyperTerminal for Win95 and WinNT

Click on the **HyperTerminal** folder within the **Programs/Accessories** folder in the start menu
Double Click on the icon called "HYPERTRM" or "HYPERTRM.EXE". This will execute the program and a dialog box called Connection Description should appear. In this dialog box it should say New Connection. In the edit box labeled **Name** enter *Model 580*. Then click **OK.**
Another dialog box called Phone Number should appear. The only edit box that needs to be changed is labeled **Connect Using.** From the list, selected by clicking on down arrow on the side of the edit box, pick from the *Direct to Comm* choices, the communications port you are planning to connect the Model 580 to. Then click **OK.**
A dialog box called Comm # Properties (# being the communications port you selected) should appear. These are the communications settings that should be set for communications with the Model 580.

Bits per second	9600	(Also called BAUD RATE)
Data bits	8	
Stop bits	1	
Flow Control	NONE	(Also called handshaking)

Once the above properties have been entered and set into the appropriate edit boxes, click **OK**
At this point you should have the main HyperTerminal program window on the screen. On the left side of the status bar which appears at the bottom of the screen, it should read "connected" with a timer counting up.
From the **FILE** menu select **SAVE**. This will save the properties you just entered for later use. The file will be named *Model 580* or *Model 580.ht* and can be used to start HyperTerminal by just double clicking on this file in the HyperTerminal folder.

Procedure for setting up Thermo Environmental Instruments' Model 580

To setup the Model 580 to communicate with the HyperTerminal or any other communications program see section 2.7.4 in the Manual. The Model 580 should be set to use *PRINTER FORMAT* with the baud rate set to *9600*. A basic understanding of the Model 580's menu system will be helpful.

To transfer data between HyperTerminal and the Model 580

HyperTerminal should be setup and the Model 580 should be set up and running. Before transferring the data, HyperTerminal or the communications program you are using should be set to capture the text being transmitted by the model 580.

To turn on the capture text feature in HyperTerminal

1 Select **CAPTURE TEXT** from the **TRANSFER** menu. A dialog box will appear asking for a file name to which the data will be written.
2. Enter the directory and filename in the edit box and then click **START**. The capture file is now open and ready to receive data.
3. Use the **BROWSE** button to open the appropriate file and directory into which you wish the captured data to be saved. Using the **BROWSE** feature only allows you to open an existing file and it will not let you create a new file.

Connect the communication cable between the selected communication port on the computer and the Model 580.
Once the Model 580 is setup and connected select Output Logged Data on the Model 580 and data should appear in the HyperTerminal Window. This is the logged data and even though it may scroll off screen all the data is being captured into the above capture file described above.
Once the data is transferred, close the capture text file. This is done in HyperTerminal by selecting **STOP** from the **TRANSFER/CAPTURE TEXT** menu.
This capture text file can now be printed using any text editor or imported into a spread sheet program for data analysis.

Thermo Environmental Instruments provides spare parts and servicing from the following locations:

Thermo Environmental Instruments Inc.
8 West Forge Parkway
Franklin, Massachusetts 02038
Telephone: (508) 520-0430
Facsimile: (508) 520-1460

Thermo Environmental Instruments Inc.
325 E. Arrow Hwy. #506
San Dimas, CA 91773
Telephone: (909) 394-2373
Facsimile: (909) 394-2367

Thermo Environmental Instruments has additional service personnel located throughout the country. Contact either service center for more information.

YSI® 556 MPS
Multi Probe System
Operations Manual

YSI Environmental

Pure Data for a Healthy Planet.™

YSI 556 MPS
Multi Probe System

Operations Manual

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1. Safety

1.1 General Safety Information

Read all safety information in this manual carefully before using the YSI 556 Multi-Probe System (MPS). Reagents that are used to calibrate and check this instrument may be hazardous to your health. Take a moment to review *Appendix D Health and Safety*.

⚠ **WARNING**

Warnings are used in this manual when misuse of the instrument could result in death or serious injury to a person.

⚠ **CAUTION**

Cautions are used in this manual when misuse of the instrument could result in mild or serious injury to a person and/or damage to equipment.

⚠ **IMPORTANT SAFETY INSTRUCTIONS!**

⚠ **SAVE THESE INSTRUCTIONS!**

⚠ In essence, the most important safety rule for use of the YSI 556 MPS is to utilize the instrument ONLY for purposes documented in this manual. This is particularly true of the YSI 6117 rechargeable battery pack that contains nickel metal hydride (NiMH) batteries. The user should be certain to read all of the safety precautions outlined below before using the instrument.

⚠ YSI 6117 Rechargeable Battery Pack Safety Information

⚠ **Restrictions on Usage**

1. Never dispose of the battery pack in a fire.
2. Do not attempt to disassemble the YSI 6117 battery pack.
3. Do not tamper with any of the electronic components or the batteries within the battery pack. Tampering with either the electronic circuitry or the batteries will result in the voiding of the warranty and the compromising of the system performance, but, more importantly, can cause safety

hazards which result from overcharging such as overheating, venting of gas, and loss of corrosive electrolyte.

4. Do not charge the battery pack outside the 0–40°C temperature range.
5. Do not use or store the battery at high temperature, such as in strong direct sunlight, in cars during hot weather, or directly in front of heaters.
6. Do not expose the battery pack to water or allow the terminals to become damp.
7. Avoid striking or dropping the battery pack. If the pack appears to have sustained damage from these actions or malfunctions after an impact or drop, the user should not attempt to repair the unit. Instead, contact YSI Customer Service. Refer to *Appendix E Customer Service*.
8. If the battery pack is removed from the YSI 556 MPS, do not store it in pockets or packaging where metallic objects such as keys can short between the positive and negative terminals.

⚠ Precautions for Users with Small Children

Keep the battery pack out of reach of babies and small children.

⚠ Danger Notifications – Misuse creates a STRONG possibility of death or serious injury.

FAILURE TO CAREFULLY OBSERVE THE FOLLOWING PROCEDURES AND PRECAUTIONS CAN RESULT IN LEAKAGE OF BATTERY FLUID, HEAT GENERATION, BURSTING, AND SERIOUS PERSONAL INJURY.

1. Never dispose of the battery pack in a fire or heat it.
2. Never allow the positive and negative terminals of the battery pack to become shorted or connected with electrically conductive materials. When the battery pack has been removed from the YSI 556 MPS, store it in a heavy plastic bag to prevent accidental shorting of the terminals.

3. Never disassemble the battery pack and do not tamper with any of the electronic components or the batteries within the battery pack. The battery pack is equipped with a variety of safety features. Accidental deactivation of any of these safety features can cause a serious hazard to the user.
4. The NiMH batteries in the battery pack contain a strong alkaline solution (electrolyte). The alkaline solution is extremely corrosive and will cause damage to skin or other tissues. If any fluid from the battery pack comes in contact with a user's eyes, immediately flush with clean water and consult a physician immediately. The alkaline solution can damage eyes and lead to permanent loss of eyesight.

⚠ Warning Notifications – Misuse creates a possibility of death or serious injury

1. Do not allow the battery pack to contact freshwater, seawater, or other oxidizing reagents that might cause rust and result in heat generation. If a battery becomes rusted, the gas release vent may no longer operate and this failure can result in bursting.
2. If electrolyte from the battery pack contacts the skin or clothing, thoroughly wash the area immediately with clean water. The battery fluid can irritate the skin.

⚠ Caution Notifications – Misuse creates a possibility of mild or serious injury or damage to the equipment.

1. Do not strike or drop the battery pack. If any impact damage to the battery pack is suspected, contact YSI Customer Service. Refer to *Appendix E Customer Service*.
2. Store the battery pack out of reach of babies and small children.
3. Store the battery pack between the temperatures of -20 and 30°C.
4. Before using the battery pack, be sure to read the operation manual and all precautions carefully. Then store this information carefully to use as a reference when the need arises.

⚠ YSI 616 Cigarette Lighter Charger Safety Information

1. This section contains important safety and operating instructions for the YSI 556 MPS cigarette lighter battery charger (YSI 616; RadioShack Number 270-1533E). BE SURE TO SAVE THESE INSTRUCTIONS.
2. Before using the YSI 616 cigarette lighter charger, read all instructions and cautionary markings on battery charger, battery pack, and YSI 556 MPS.
3. Charge the YSI 6117 battery pack with the YSI 616 cigarette lighter charger ONLY when the YSI 6117 is installed in the YSI 556 MPS.
4. Do not expose charger to rain, moisture, or snow.
5. Use of an attachment not recommended or sold by the battery charger manufacturer may result in a risk of fire, electric shock, or injury to persons.
6. To reduce risk of damage to cigarette lighter and cord, pull by cigarette lighter rather than cord when disconnecting charger.
7. Make sure that the cord is located so that it will not be stepped on, tripped over, or otherwise subjected to damage or stress.
8. Do not operate charger with damaged cord or cigarette lighter connector – replace it immediately.
9. Do not operate charger if it has received a sharp blow, been dropped, or otherwise damaged in any way; contact YSI Customer Service. Refer to *Appendix E Customer Service*.
10. Do not disassemble charger other than to change the fuse as instructed. Replace the part or send it to YSI Product Service if repair is required (refer to *Appendix E Customer Service*). Incorrect reassembly may result in a risk of electric shock or fire.
11. To reduce risk of electric shock, unplug charger before attempting any maintenance or cleaning. Turning off controls will not reduce this risk.

⚠ YSI 556 MPS Water Leakage Safety Information

The YSI 556 MPS has been tested and shown to comply with IP67 criterion, i.e. submersion in 1 meter of water for 30 minutes with no leakage into either the battery compartment or the main case. However, if the instrument is submersed for periods of time in excess of 30 minutes, leakage may occur with subsequent damage to the batteries, the rechargeable battery pack circuitry, and/or the electronics in the main case.

If leakage into the battery compartment is observed when using alkaline C cells, remove batteries, dispose of batteries properly, and dry the battery compartment completely, ideally using compressed air. If corrosion is present on the battery terminals, contact YSI Customer Service for instructions. Refer to *Appendix E Customer Service*.

If leakage into the battery compartment is observed when using the YSI 6117 rechargeable battery pack, remove the battery assembly and set aside to dry. Return the battery pack to YSI Product Service for evaluation of possible damage. Finally dry the battery compartment completely, ideally using compressed air. If corrosion is present on the battery terminals, contact YSI Customer Service for instructions. Refer to *Appendix E Customer Service*.

⚠ **CAUTION:** If water has contacted the rechargeable battery pack, do not attempt to reuse it until it has been evaluated by YSI Product Service (refer to *Appendix E Customer Service*). Failure to follow this precaution can result in serious injury to the user.

If it is suspected that leakage into the main cavity of the case has occurred, remove the batteries immediately and return the instrument to YSI Product Service for damage assessment. Refer to *Appendix E Customer Service*.

⚠ **CAUTION:** Under no circumstances should the user attempt to open the main case.

2. General Information

2.1 Description

The rugged and reliable YSI 556 MPS (Multi-Probe System) combines the versatility of an easy-to-use, easy-to-read handheld unit with all the functionality of a multi-parameter system. Featuring a waterproof, impact-resistant case, the YSI 556 MPS simultaneously measures dissolved oxygen, conductivity, temperature, and optional pH and ORP. A simple cellular phone style keypad and large display make the instrument easy to use. The YSI 556 MPS is compatible with YSI EcoWatch™ for Windows™ software.

The YSI 556 MPS assists the user in conforming to Good Laboratory Practice (GLP) standards which help ensure that quality control/quality assurance methods are followed. Battery life is displayed with a fuel gauge, and the user can choose standard alkaline batteries or an optional rechargeable battery pack.

The 1.5 MB memory can store more than 49,000 data sets. Other options include a flow cell and barometer. The internal barometer can be user-calibrated and displayed along with other data, used in dissolved oxygen calibrations, and logged to memory for tracking changes in barometric pressure.

Features

- Waterproof - meets IP67 specifications
- Field-replaceable DO electrode module; pH and pH/ORP sensors
- Compatible with Ecowatch™ for Windows™ data analysis software
- Assists with Good Laboratory Practice Standards (GLP)
- Choice of DO membrane material for different applications
- Easy-to-use, screw-on cap DO membranes
- User-upgradable software from YSI website
- Three-year warranty on the instrument; one-year on the probe modules
- Available with 4,10, and 20 m cable lengths
- Stores over 49,000 data sets, time and date stamped
- Auto temperature compensating display contrast

- Optional barometer
- Optional rechargeable battery pack or standard alkaline batteries

2.2 Unpacking the Instrument

1. Remove the instrument from the shipping box. Note that the probe module and sensors are shipped in a separate box and will be unpacked later in Section *3.2 Unpacking the Probe Module*.

   NOTE: Do not discard any parts or supplies.

2. Use the packing list to ensure all items are present.

3. Visually inspect all components for damage.

   NOTE: If any parts are missing or damaged, contact your YSI Service Center immediately. Refer to *Appendix E Customer Service* or www.ysi.com.

2.3 Features of the YSI 556 Multi-Probe System

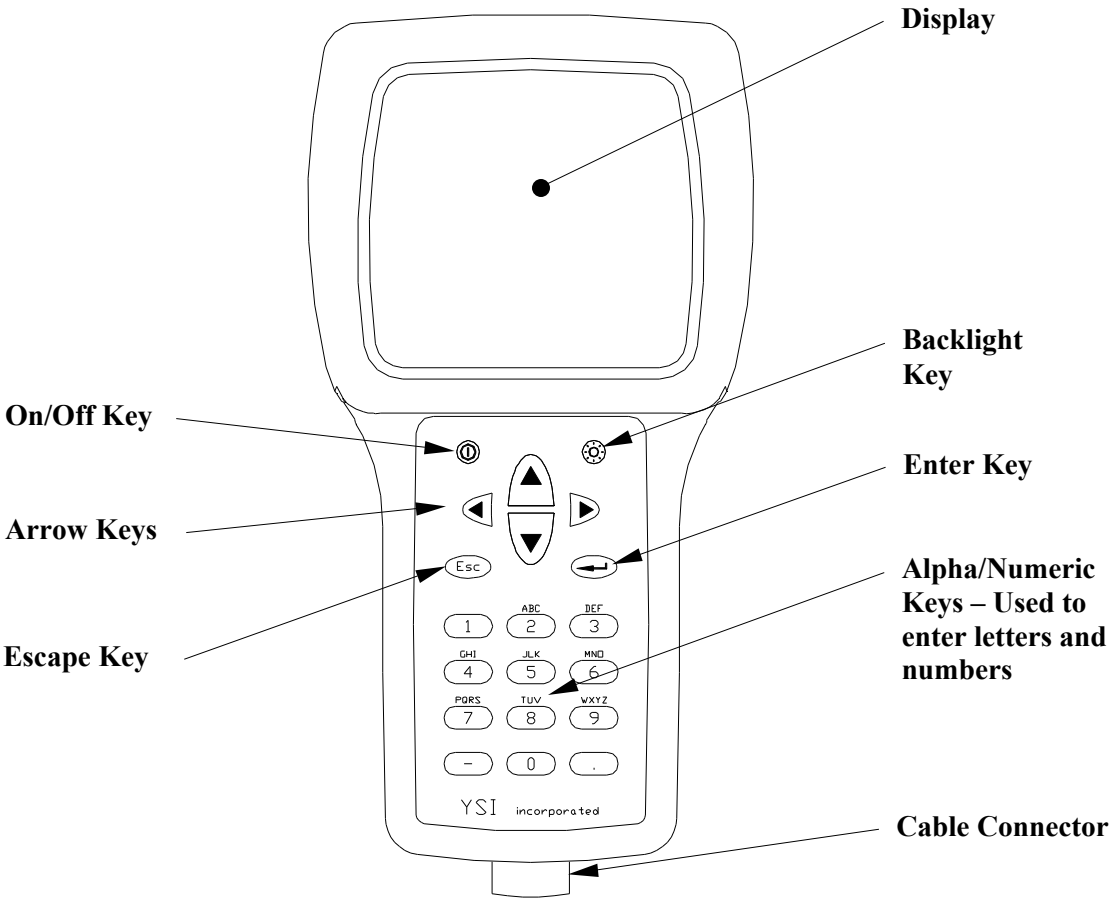


Figure 2.1 Front View of YSI 556 MPS

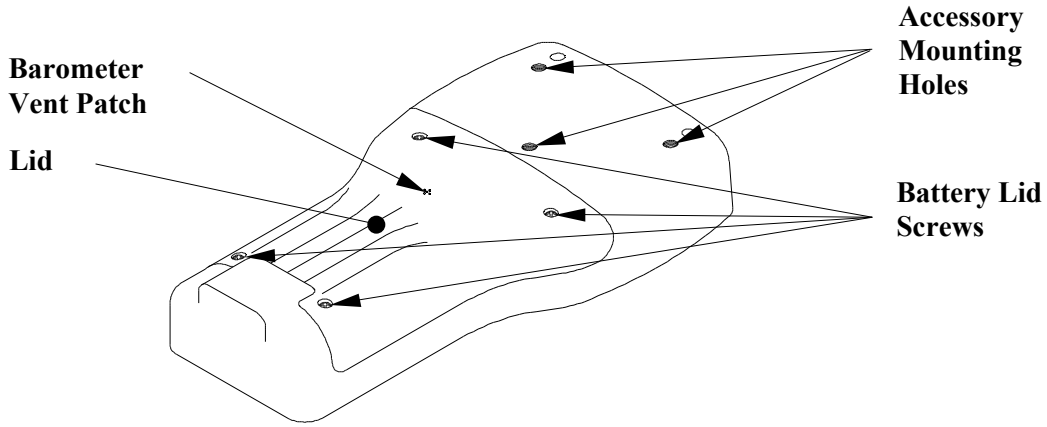


Figure 2.2 Back View of YSI 556 MPS

2.4 Batteries

2.4.1 Battery Life

Standard Alkaline Batteries

With the standard battery configuration of 4 alkaline C cells, the YSI 556 MPS will operate continuously for approximately 180 hours. Assuming a standard usage pattern when sampling of 3 hours of "on time" in a typical day, the alkaline cells will last approximately 60 days.

Optional Rechargeable Battery Pack

When fully charged, the optional rechargeable battery pack will provide approximately 50 hours of battery life.

2.4.2 Inserting 4 C Batteries

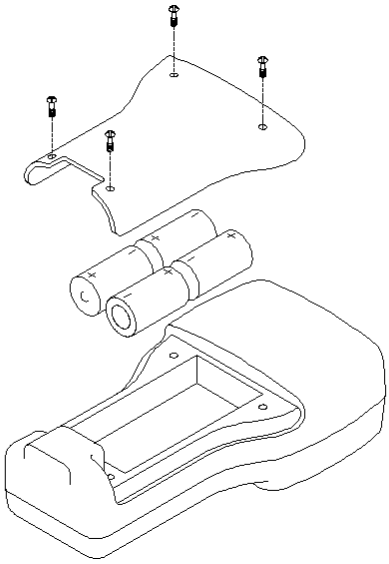


Figure 2.3 Inserting C Cells

⚠ **CAUTION: Install batteries properly to avoid damage to the instrument.**

1. Loosen the four screws in the battery lid on the back of the instrument using any screwdriver.

2. Remove the battery lid.

3. Insert four C batteries between the clips following the polarity (+ and -) labels on the bottom of the battery compartment.

4. Check gasket for proper placement on the battery lid.

5. Replace the battery lid and tighten the 4 screws securely and evenly.

   NOTE: Do not over-tighten the screws.

2.4.3 Inserting Optional Rechargeable Battery Pack

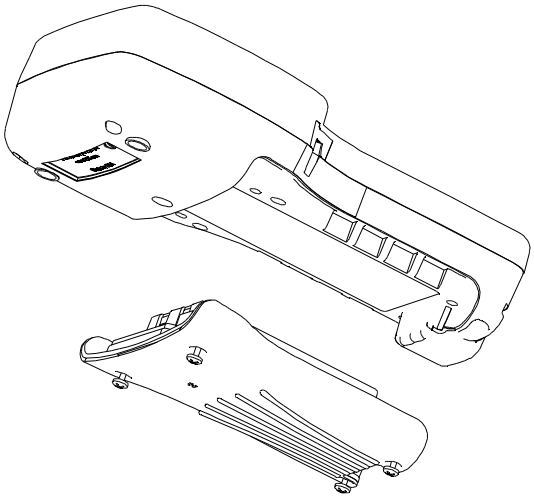


Figure 2.4 Inserting Battery Pack

⚠ **CAUTION: Read all cautions and warnings that come with the battery pack *before* using the battery pack.**

1. Loosen the four screws in the battery lid on the back of the instrument using any screwdriver.

2. Remove the C battery lid and store for future use. Remove C batteries, if installed.

3. Check for proper placement of gasket on the rechargeable battery pack and lid.

4. Install the rechargeable battery pack and lid and tighten the 4 screws securely and evenly.

   NOTE: Do not over tighten the screws.

2.4.4 Charging the Optional Rechargeable Battery Pack

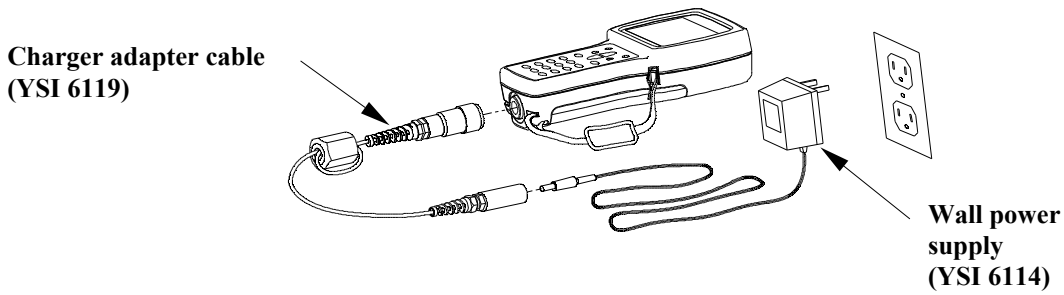


Figure 2.5 Charging the Battery Pack

⚠ **CAUTION: Do not use or store the battery pack at extreme temperatures such as in strong direct sunlight, in cars during hot weather or close to heaters.**

1. Install the rechargeable battery pack into the instrument as described in Section *2.4.3 Inserting Optional Rechargeable Battery Pack*.

2. Attach the charger adapter cable (YSI 6119) to the instrument.

   NOTE: Wall power supplies for use in countries outside the US and Canada can be found in *Appendix B Instrument Accessories*.

3. Insert the barrel connector of the wall power supply into the barrel of the adapter cable.

⚠ **CAUTION: Do not charge the battery pack continuously for more than 48 hours.**

⚠ **CAUTION: Do not drop or expose to water.**

⚠ **CAUTION: Do not charge the battery pack at temperatures below 0°C or above 40°C.**

4. Plug the wall power supply into an AC power outlet for approximately 2 hours to obtain an 80% to 90% charge and for 6 hours to get a full charge.

   NOTE: The battery pack can be recharged whether the instrument is on or off.

2.4.5 Storing the Battery Pack

Remove the battery pack from the instrument when the instrument will not be used for extended periods of time to prevent over discharge of the battery pack.

Store the battery pack in a heavy plastic bag to prevent accidental shorting of the terminals. Store between –20 and 30°C.

2.4.6 Optional Cigarette Lighter Charger

⚠ **CAUTION: Read all warnings and cautions that come with the charger before using the charger.**

⚠ **CAUTION: Only use cigarette lighter charger when *rechargeable* battery pack is inserted into instrument.**

⚠ **CAUTION: Do not mishandle cigarette lighter charger. Do not expose to moisture.**

1. Plug the barrel connector of the cigarette lighter charger into the mating end of the YSI 6119 Charger Adapter Cable.

2. Attach the MS-19 end of the YSI 6119 Charger Adapter Cable to the instrument.

3. Make one of the following modifications to the other end of the charger:

   Slide the adapter ring off the plug to use the device with an American or Japanese vehicle.

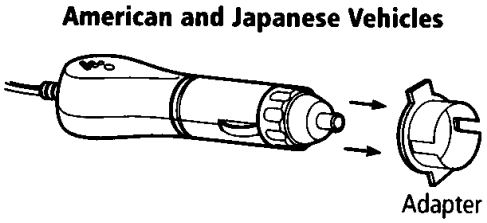


Figure 2.6 Charger Plug Adapter Use

Leave the adapter ring on the plug and position it so that the slots on the adapter ring line up with the plug's spring clips to use the device on a European vehicle.

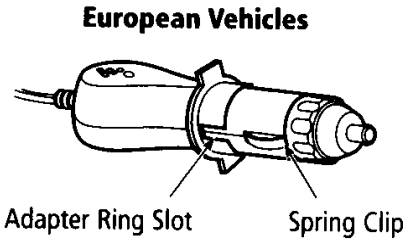


Figure 2.7 European Charger Plug Adapter Use

NOTE: If the charger stops working properly, refer to Section *13 Troubleshooting*.

2.5 Power On

Press and release the on/off button in the upper left corner of the instrument keypad to turn the instrument on or off. See Figure 2.1 Front View of YSI 556 MPS.

2.6 Setting Display Contrast

The display contrast automatically compensates for temperature changes. However, under extreme temperature conditions you may wish to optimize the display by manual adjustment as follows:

1. Press and *hold down* the backlight key in the upper right corner of the keypad and press the “up” arrow to increase (darken) the contrast.

2. Press and *hold down* the backlight key in the upper right corner of the keypad and press the “down” arrow to decrease (lighten) the contrast.

2.7 Backlight

Press and *release* the backlight key in the upper right corner of the keypad to turn the backlight on or off. See Figure 2.1 Front View of YSI 556 MPS.

NOTE: The backlight turns off automatically after two minutes of non-use.

2.8 General Screen Features

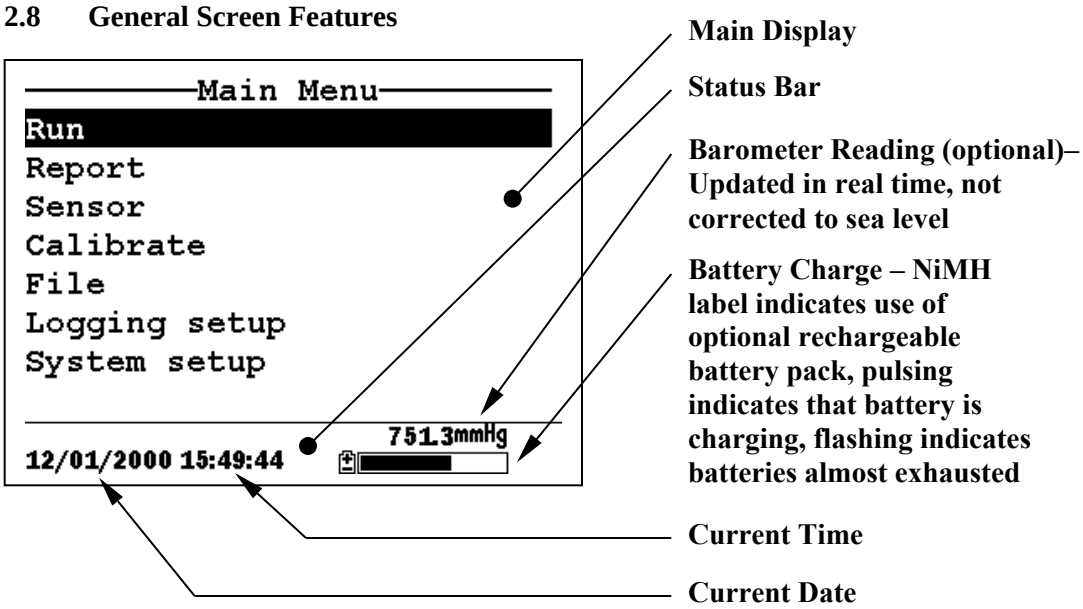


Figure 2.8 Main Menu Screen

2.9 Keypad Use

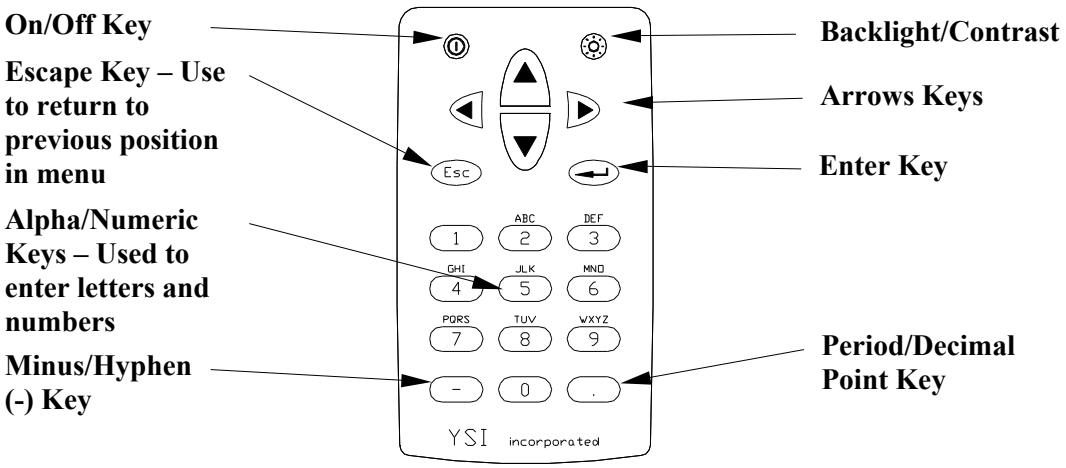


Figure 2.9 Keypad Features

KEY	LETTER/ NUMBER
1	1
2	ABC2abc3
3	DEF3def3
4	GHI4ghi4
5	JKL5jkl5
6	MNO6mno6
7	PQRS7pqrs7
8	TUV8tuv8
9	WXYZ9wxyz9
0	0

Figure 2.10 Keypad Letters & Numbers

1. See Figure 2.10 Keypad Letters & Numbers and press the appropriate key repeatedly until letter or number desired appears in display.

   NOTE: Press the key repeatedly in rapid succession to get to the desired letter or number. If you pause for more than a

second, the cursor automatically scrolls to the right to prepare for the next input.

EXAMPLE 1: Press the **6** key *once* and *release* to display an uppercase "M."

EXAMPLE 2: Press the **6** key *four times* and *release* to display the number "6."

EXAMPLE 3: Press the **6** key *five times* and *stop* to display a lowercase "m."

2. Press the left arrow key to go back and reenter a number or letter that needs to be changed.

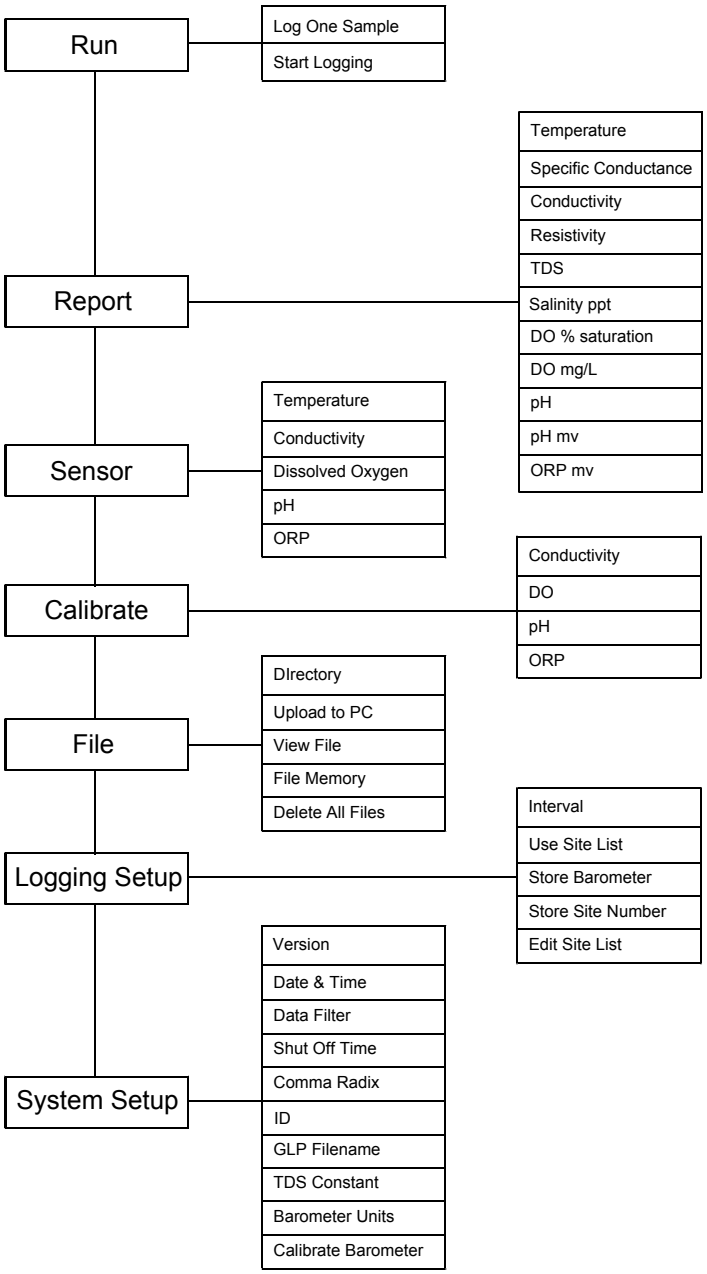
3. Press the **Enter** key when your entry is complete.

   NOTE: The instrument software permits only numeric entries in many instances, such as when setting the clock or entering calibration parameters.

2.10 Instrument Reset

The YSI 556 MPS is characterized by sophisticated software that should provided trouble-free operation. However as with all high-capability software packages, it is always possible that the user will encounter circumstances in which the instrument does not respond to keypad entry. If this occurs, the instrument function can easily be restored by removing and then reapplying battery power. Simply remove either your C-cells or rechargeable battery pack from the battery compartment, wait 30 seconds and then replace the batteries. See Section *2.4 Batteries* for battery removal/reinstallation instructions.

2.11 Menu Flowchart



3. Probe Module

3.1 Introduction

The YSI 5563 Probe module is used for measuring dissolved oxygen, temperature, conductivity, and optional pH and ORP. The probe module is rugged, with the sensors enclosed in a heavy duty probe sensor guard with attached sinking weight. A 4, 10 or 20 meter cable is directly connected to the probe module body making it waterproof. An MS-19 connector at the end of the cable makes the YSI 5563 fully compatible with the YSI 556 Multi-Probe System.

3.2 Unpacking the Probe Module

1. Remove the YSI 5563 Probe module from the shipping boxes.

   NOTE: Do not discard any parts or supplies.

2. Use the packing list to ensure all items are present.

3. Visually inspect all components for damage.

   NOTE: If any parts are missing or damaged, contact your YSI Service Center immediately. Refer to *Appendix E Customer Service* or www.ysi.com.

3.3 Features of the YSI 5563 Probe Module

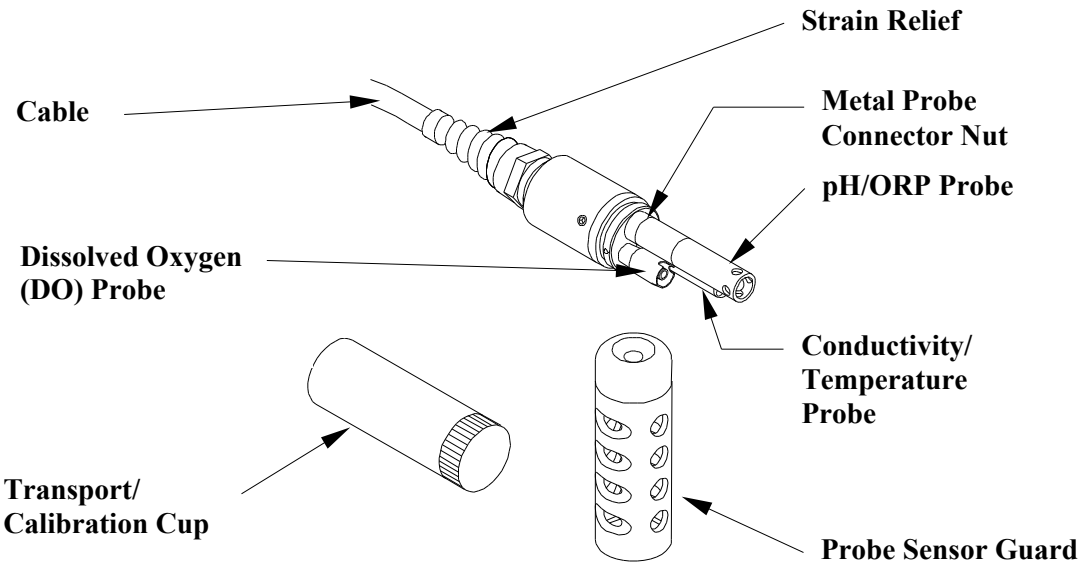


Figure 3.1 Probe Module

3.4 Preparing the Probe Module

To prepare the probe module for calibration and operation, you need to install the sensors into the connectors on the probe module bulkhead. In addition to sensor installation, you need to install a new DO membrane cap.

3.4.1 Sensor Installation

Whenever you install, remove or replace a sensor, it is extremely important that the entire probe module and all sensors be thoroughly dried prior to the removal of a sensor or a sensor port plug. This will prevent water from entering the port. Once you remove a sensor or plug, examine the connector inside the probe module sensor port. If any moisture is present, use compressed air to completely dry the connector. If the connector is corroded, return the probe module to your dealer or directly to YSI Customer Service. Refer to *Appendix E Customer Service*.

Conductivity/Temperature and pH, pH/ORP Sensor Installation

1. Unscrew and remove the probe sensor guard.

2. Using the sensor installation tool supplied in the YSI 5511 maintenance kit, unscrew and remove the sensor port plugs.

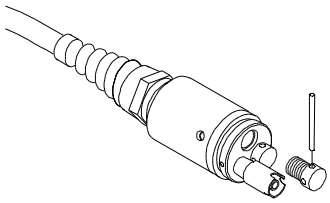


Figure 3.2 Port Plug Removal

3. Locate the port with the connector that corresponds to the sensor that is to be installed.

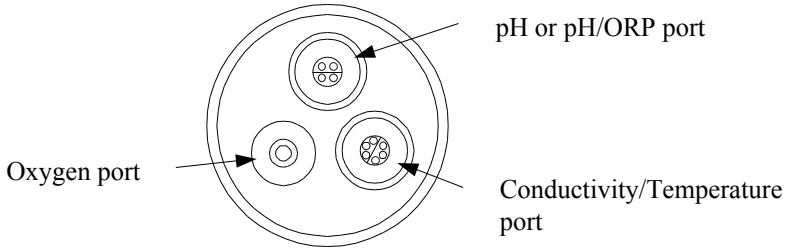


Figure 3.3 Sensor Port Identification

4. Apply a thin coat of o-ring lubricant (supplied in the YSI 5511 maintenance kit) to the o-rings on the connector side of the sensor (see Figure 3.4 O-Ring Lubrication).

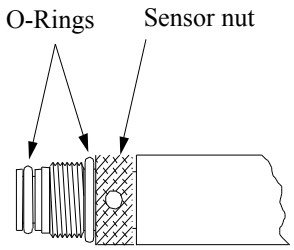


Figure 3.4 O-Ring Lubrication

⚠ **CAUTION:** Make sure that there are NO contaminants between the O-ring and the sensor. Contaminants that are present under the O-ring may cause the O-ring to leak.

5. Be sure the probe module sensor port is free of moisture and then insert the sensor into the correct port. Gently rotate the sensor until the two connectors align.

6. With connectors aligned, screw down the sensor nut using the sensor installation tool.

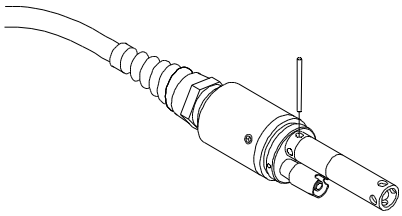


Figure 3.5 Sensor Installation

⚠ **CAUTION:** Do not cross thread the sensor nut. Tighten the nut until it is flush with the face of the probe module bulkhead. Do not over tighten.

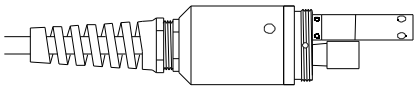


Figure 3.6 Bulkhead Seating

7. Repeat steps 3-6 for any other sensors.

8. Replace the probe sensor guard.

Dissolved Oxygen Sensor Installation

The YSI 5563 comes with the DO sensor already installed. Refer to Section *11.1.2 DO Sensor Replacement* for instructions on installing the YSI 558 Replaceable DO Module Kit.

3.4.2 Membrane Cap Selection

The YSI 5563 is shipped with a YSI 5909 kit that contains membrane caps made with 2 mil polyethylene (PE), a material which should be ideal for most field applications of the 556. However, YSI also offers membrane caps made with two other materials (1 mil polyethylene and 1 mil Teflon) which some users may also prefer. All membranes available for the 556/5563 system provide comparable accuracy if used properly. The difference between the two thicknesses of PE is found in the trade-off of flow dependence and response time as described below. Teflon is offered because some users may prefer to continue using the traditional membrane material used by YSI. To avoid confusion, the membrane caps are color coded as described below and can be ordered in kits as noted:

1 mil Teflon – Black Caps (Kit = YSI 5906)
1 mil Polyethylene (PE) – Yellow Caps (Kit = YSI 5908)
2 mil Polyethylene (PE) – Blue Caps (Kit = YSI 5909)

The 1 mil Teflon caps will offer traditional, reliable performance for most dissolved oxygen applications. The 1 mil PE caps will provide a significantly faster dissolved oxygen response (as long as your 556 Data Filter is set correctly as described below in Sections 10.2 and 10.3.1)) while also giving readings which are significantly less flow dependent than the 1 mil Teflon caps. Finally, 2 mil PE caps will show a large reduction in flow dependence over 1 mil Teflon while not significantly increasing the response time. Generally, one of the PE caps is likely to provide better performance for your application.

IMPORTANT: No matter which type of membrane cap you select, you will also have to confirm your selection in the 556

software from the Sensor menu as described in Section *4 Sensors*.

3.4.3 Membrane Cap Installation

NOTE: The YSI 5563 DO sensor (already installed in the probe module) was shipped dry. A shipping membrane was installed to protect the electrode. **A new membrane cap must be installed before the first use**.

1. Unscrew and remove the probe sensor guard.
2. Unscrew, remove, and discard the old membrane cap.
3. Thoroughly rinse the sensor tip with distilled water.
4. Prepare the electrolyte according to the directions on the electrolyte solution bottle.
5. Hold the new membrane cap and fill it at least 1/2 full with the electrolyte solution.
6. Screw the membrane cap onto the sensor moderately tight. A small amount of electrolyte should overflow.

⚠ **CAUTION:** Do not touch the membrane surface.

7. Screw the probe sensor guard on moderately tight.

3.5 Transport/Calibration Cup

The YSI 5563 Probe module has been supplied with a convenient transport/calibration cup. This cup is an ideal container for calibration of the different sensors, minimizing the amount of solution needed. Refer to Section *6 Calibrate*.

3.5.1 Transport/Calibration Cup Installation

1. Remove probe sensor guard, if already installed.

2. Ensure that an o-ring is installed in the o-ring groove on the threaded end of the probe module body.

3. Screw the transport/calibration cup on the threaded end of the probe module and securely tighten.

   NOTE: Do not overtighten as this could cause damage to the threaded portions.

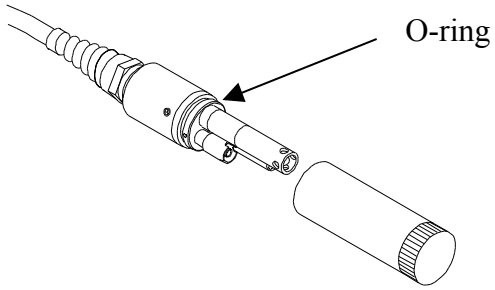


Figure 3.7 Transport/Calibration Cup Installation

3.6 Instrument/Cable Connection

Attach the cable to the instrument as follows:

1. Line up the pins and guides on the cable with the holes and indentations on the cable connector at the bottom of the YSI 556 instrument. See Figure 2.1 Front View of YSI 556 MPS.

2. Holding the cable firmly against the cable connector, turn the locking mechanism clockwise until it snaps into place.

Remove the cable from the instrument by turning the cable connector counterclockwise until the cable disengages from the instrument.

The Sensors Enabled screen allows the user to enable or disable each of the sensors and select which membrane material will be used for the dissolved oxygen sensor. Disabled sensors will not be displayed on the screen in real time or logged to files.

1. Press the **On/off** key to display the run screen.

2. Press the **Escape** key to display the main menu screen.

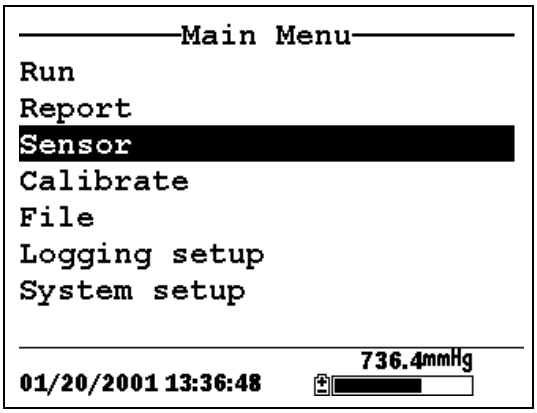


Figure 4.1 Main Menu Screen

3. Use the arrow keys to highlight the **Sensor** selection.

4. Press the **Enter** key to display the sensors enabled screen.

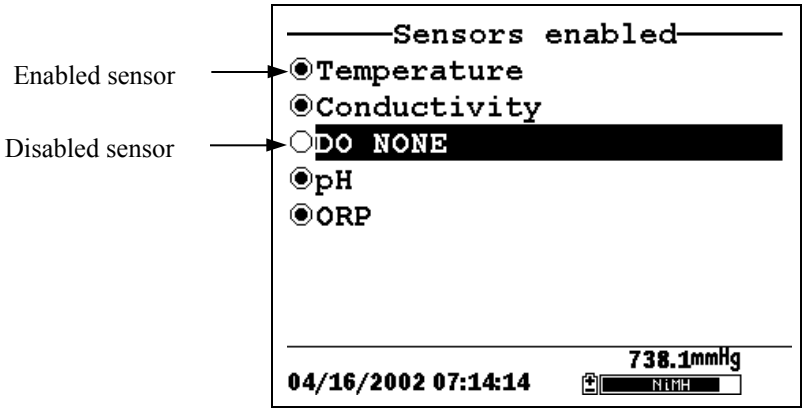


Figure 4.2 Sensors Enabled Screen Before DO Membrane Selection

A black dot to the left of a sensor indicates that sensor is enabled. Sensors with an empty circle are disabled.

Highlight the “DO None” entry as shown above and press **Enter** to display the membrane choice screen. Consult Section *3.4.2 Membrane Cap Selection* for information on the advantages of each type of membrane material. Blue membrane caps using 2 mil polyethylene (PE) were shipped with your YSI 5563 and are likely to be the best choice for most 556 field applications.

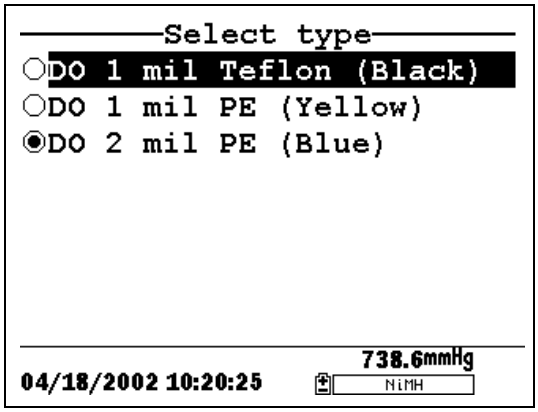


Figure 4.3 Membrane Selection Screen

Highlight the desired membrane choice – in this case, 2 mil PE -- and press Enter to activate your selection with a dot to the left of the screen. Then press **Escape** to return to the Sensor menu that now shows your DO membrane selection.

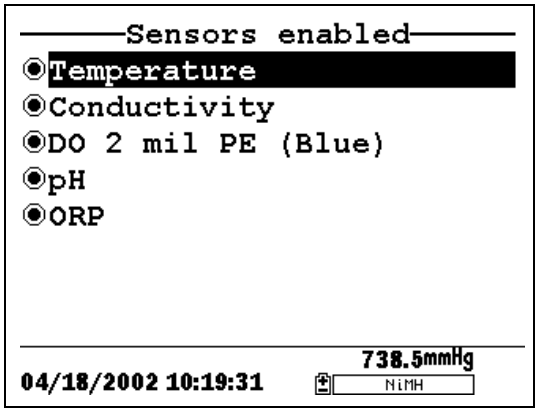


Figure 4.4 Sensors Enabled Screen After DO Membrane Selection

NOTE: The Temperature sensor cannot be disabled. Most other sensors require temperature compensation for accurate readings. In addition, the conductivity sensor must be activated in order to obtain accurate dissolved oxygen mg/L readings.

5. Use the arrow keys to highlight the sensor you want to change, then press the **Enter** key to enable or disable it.

6. Repeat step 5 for each sensor you want to change.

7. Press the **Escape** key to return to the main menu screen.

5. Report

The Report Setup screen allows the user to select which sample parameters and units the YSI 556 MPS will display on the screen. It does NOT determine which parameters are logged to memory. Refer to Section *4 Sensors*.

1. Press the **On/off** key to display the run screen.

2. Press the **Escape** key to display the main menu screen.

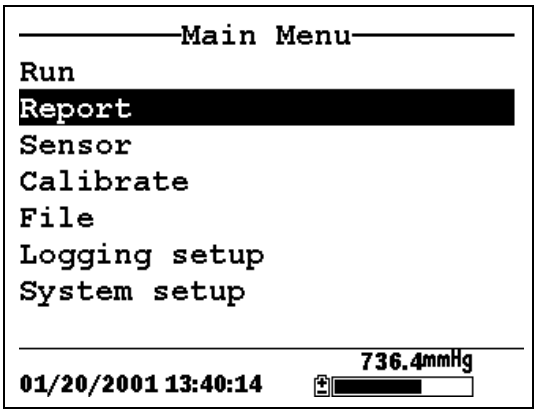


Figure 5.1 Main Menu

3. Use the arrow keys to highlight the **Report** selection.

4. Press the **Enter** key to display the report setup screen.

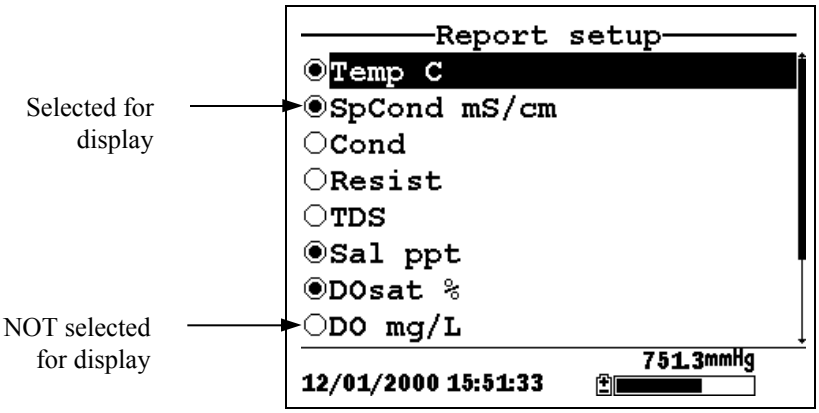


Figure 5.2 Report Setup Screen

NOTE: A black dot to the left of a parameter indicates that parameter is selected for display. Parameters with an empty circle will not be displayed.

NOTE: You may have to scroll down past the bottom of the screen to see all the parameters.

5. Use the arrow keys to highlight the parameter you want to change, then press the **Enter** key. If you can't find the parameter you want, even after scrolling down past the bottom of the screen, the sensor used for that parameter is disabled. Refer to Section *4 Sensors*.

6. If you selected Temperature, Specific Conductivity, Conductivity, Resistance or Total Dissolved Solids, the Units screen will appear.

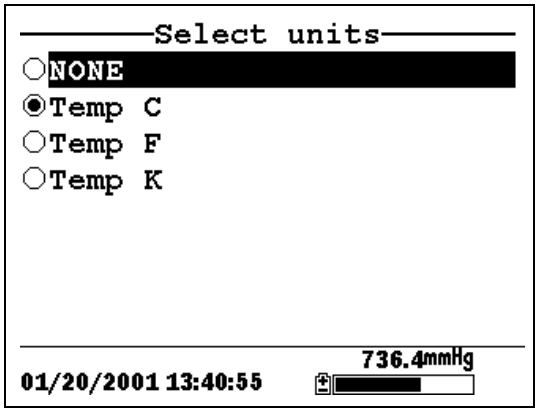


Figure 5.3 Units Screen

7. Use the arrow keys to select the units desired, then press the **Enter** key to return to the report setup screen.

   If you selected Salinity, Dissolved Oxygen %, Dissolved Oxygen mg/L, pH, pH mv or ORP mv, the selection dot will simply toggle on or off.

8. Repeat steps 5 and 6 for each parameter you want to change.

NOTE: All parameters may be enabled at the same time.

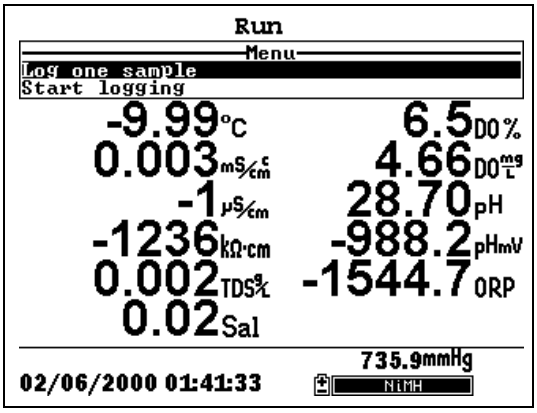


Figure 5.4 All Parameters Displayed

9. Press the **Escape** key to return to the Main menu screen.

6. Calibrate

All of the sensors, except temperature, require periodic calibration to assure high performance. You will find specific calibration procedures for all sensors that require calibration in the following sections. If a sensor listed is not installed in your probe module, skip that section and proceed to the next sensor until the calibration is complete.

⚠ **CAUTION:** Reagents that are used to calibrate and check this instrument may be hazardous to your health. Take a moment to review *Appendix D Health and Safety*. Some calibration standard solutions may require special handling.

6.1 Getting Ready to Calibrate

6.1.1 Containers Needed to Calibrate the Probe Module

The transport/calibration cup that comes with your probe module serves as a calibration chamber for all calibrations and minimizes the volume of calibration reagents required.

Instead of the transport/calibration cup, you may use laboratory glassware to perform calibrations. If you do not use the transport/calibration cup that is designed for the probe module, you are cautioned to do the following:

- Perform all calibrations with the Probe Sensor Guard installed. This protects the sensors from possible physical damage.
- Use a ring stand and clamp to secure the probe module body to prevent the module from falling over. Most laboratory glassware has convex bottoms.
- Ensure that all sensors are immersed in calibration solutions. Many of the calibrations factor in readings from other sensors (e.g., temperature sensor). The top vent hole of the conductivity sensor must also be immersed during some calibrations.

6.1.2 Calibration Tips

1. If you use the Transport/Calibration Cup for dissolved oxygen (DO) calibration, make certain to loosen the seal to allow pressure equilibration before calibration. The DO calibration is a water-saturated air calibration.
2. The key to successful calibration is to ensure that the sensors are completely submersed when calibration values are entered. Use recommended volumes when performing calibrations.
3. For maximum accuracy, use a small amount of previously used calibration solution to pre-rinse the probe module. You may wish to save old calibration standards for this purpose.
4. Fill a bucket with ambient temperature water to rinse the probe module between calibration solutions.
5. Have several clean, absorbent paper towels or cotton cloths available to dry the probe module between rinses and calibration solutions. Shake the excess rinse water off of the probe module, especially when the probe sensor guard is installed. Dry off the outside of the probe module and probe sensor guard. Making sure that the probe module is dry reduces carry-over contamination of calibrator solutions and increases the accuracy of the calibration.
6. If you are using laboratory glassware for calibration, you do not need to remove the probe sensor guard to rinse and dry the sensors between calibration solutions. The inaccuracy resulting from simply rinsing the sensor compartment and drying the outside of the guard is minimal.
7. If you are using laboratory glassware, remove the stainless steel weight from the bottom of the probe sensor guard by turning the weight counterclockwise. When the weight is removed, the calibration solutions have access to the sensors without displacing a lot of fluid. This also reduces the amount of liquid that is carried between calibrations.
8. Make certain that port plugs are installed in all ports where sensors are not installed. It is extremely important to keep these electrical connectors dry.

6.1.3 Recommended Volumes

Follow these instructions to use the transport/calibration cup for calibration procedures.

- Ensure that an o-ring is installed in the o-ring groove of the transport/calibration cup bottom cap, and that the bottom cap is securely tightened.

  NOTE: Do not over-tighten as this could cause damage to the threaded portions.

- Remove the probe sensor guard, if it is installed.
- Remove the o-ring, if installed, from the probe module and inspect the installed o-ring on the probe module for obvious defects and, if necessary, replace it with the extra o-ring supplied.
- Some calibrations can be accomplished with the probe module upright or upside down. A separate clamp and stand, such as a ring stand, is required to support the probe module in the inverted position.
- To calibrate, follow the procedures in the next section, Calibration Procedures. The approximate volumes of the reagents are specified below for both the upright and upside down orientations.
- When using the Transport/Calibration Cup for dissolved oxygen % saturation calibration, make certain that the vessel is vented to the atmosphere by loosening the bottom cap or cup assembly and that approximately 1/8” of water is present in the cup.

Sensor to Calibrate	Upright	Upside Down
Conductivity	55ml	55ml
pH/ORP	30ml	60ml

Table 6.1 Calibration Volumes

6.2 Calibration Procedures

6.2.1 Accessing the Calibrate Screen

1. Press the **On/off** key to display the run screen.
2. Press the **Escape** key to display the main menu screen.
3. Use the arrow keys to highlight the **Calibrate** selection.

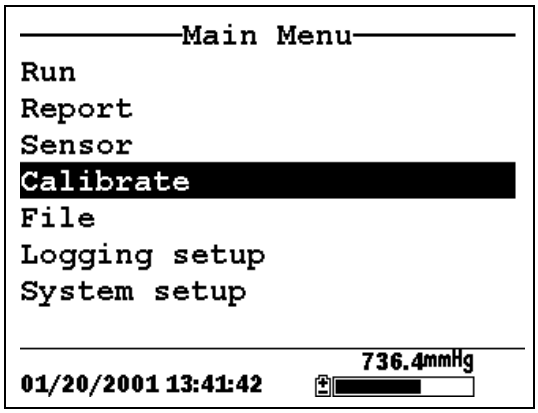


Figure 6.1 Main Menu

4. Press the **Enter** key. The Calibrate screen is displayed.

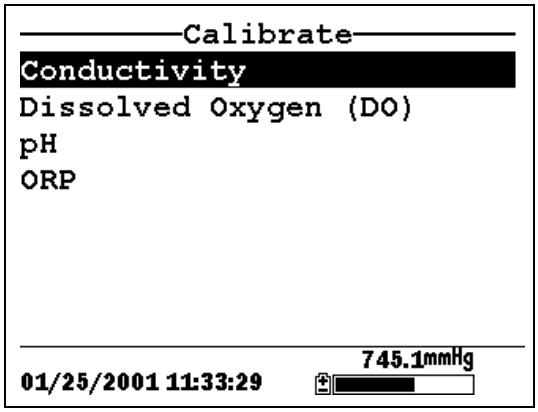


Figure 6.2 Calibrate Screen

6.2.2 Conductivity Calibration

This procedure calibrates specific conductance (recommended), conductivity and salinity. Calibrating any one option automatically calibrates the other two.

1. Go to the calibrate screen as described in Section 6.2.1 Accessing the Calibrate Screen.

2. Use the arrow keys to highlight the **Conductivity** selection. See Figure 6.2 Calibrate Screen.

3. Press **Enter.** The Conductivity Calibration Selection Screen is displayed.

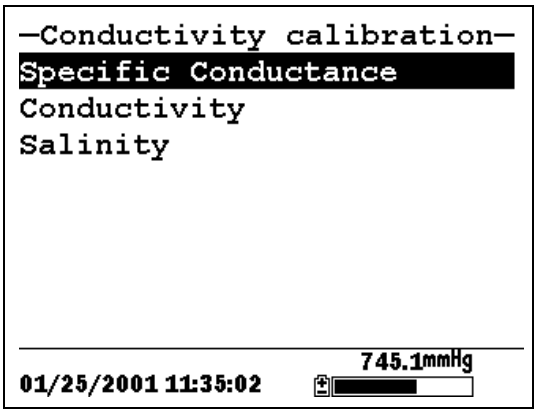


Figure 6.3 Conductivity Calibration Selection Screen

4. Use the arrow keys to highlight the Specific Conductance selection.

5. Press **Enter.** The Conductivity Calibration Entry Screen is displayed.

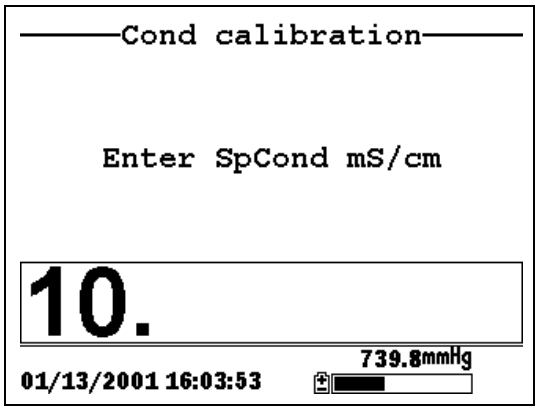


Figure 6.4 Conductivity Calibration Entry Screen

6. Place the correct amount of conductivity standard (see Table 6.1 Calibration Volumes) into a clean, dry or pre-rinsed transport/calibration cup.

⚠ **WARNING**: Calibration reagents may be hazardous to your health. See *Appendix D Health and Safety* for more information.

NOTE: For maximum accuracy, the conductivity standard you choose should be within the same conductivity range as the samples you are preparing to measure. However, we do not recommend using standards less than 1 mS/cm. For example:

- For fresh water use a 1 mS/cm conductivity standard.
- For brackish water use a 10 mS/cm conductivity standard.
- For seawater use a 50 mS/cm conductivity standard.

NOTE: Before proceeding, ensure that the sensor is as dry as possible. Ideally, rinse the conductivity sensor with a small amount of standard that can be discarded. Be certain that you avoid cross-contamination of solutions. Make certain that there are no salt deposits around the oxygen and pH/ORP sensors, particularly if you are employing standards of low conductivity.

7. Carefully immerse the sensor end of the probe module into the solution.

8. Gently rotate and/or move the probe module up and down to remove any bubbles from the conductivity cell.

   NOTE: The sensor must be completely immersed past its vent hole. Using the recommended volumes from Table 6.1 Calibration Volumes, should ensure that the vent hole is covered.

9. Screw the transport/calibration cup on the threaded end of the probe module and securely tighten.

   NOTE: Do not overtighten as this could cause damage to the threaded portions.

10. Use the keypad to enter the calibration value of the standard you are using.

    NOTE: Be sure to enter the value in **mS/cm at 25°C**.

11. Press **Enter**. The Conductivity Calibration Screen is displayed.

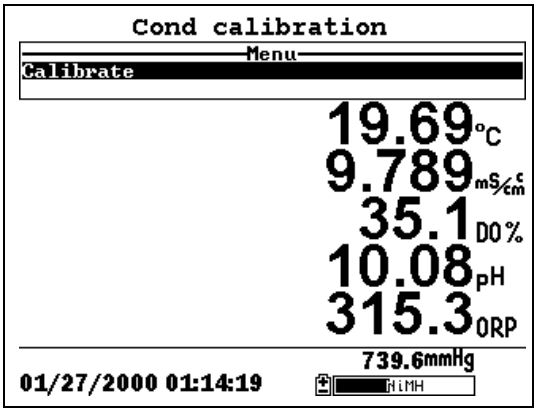


Figure 6.5 Conductivity Calibration Screen

12. Allow at least one minute for temperature equilibration before proceeding. The current values of all enabled sensors

will appear on the screen and will change with time as they stabilize.

13. Observe the reading under Specific Conductance. When the reading shows no significant change for approximately 30 seconds, press **Enter**. The screen will indicate that the calibration has been accepted and prompt you to press **Enter** again to Continue.

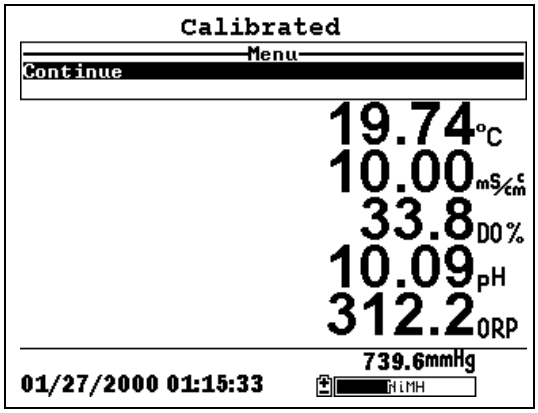


Figure 6.6 Calibrated

14. Press **Enter**. This returns you to the Conductivity Calibrate Selection Screen, See Figure 6.3 Conductivity Calibration Selection Screen.

15. Press **Escape** to return to the calibrate menu. See Figure 6.2 Calibrate Screen.

16. Rinse the probe module and sensors in tap or purified water and dry.

6.2.3 Dissolved Oxygen Calibration

This procedure calibrates dissolved oxygen. Calibrating any one option (% or mg/L) automatically calibrates the other.

1. Go to the calibrate screen as described in Section *6.2.1 Accessing the Calibrate Screen*.

NOTE: The instrument must be on for at least 20 minutes to polarize the DO sensor before calibrating.

2. Use the arrow keys to highlight the **Dissolved Oxygen** selection. See Figure 6.2 Calibrate Screen.
3. Press **Enter**. The dissolved oxygen calibration screen is displayed.

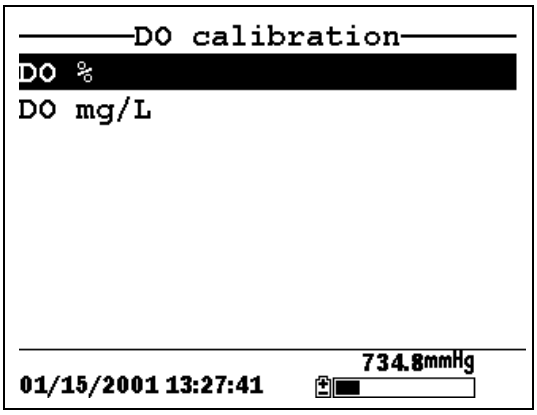


Figure 6.7 DO Calibration Screen

DO Calibration in % Saturation

1. Use the arrow keys to highlight the DO% selection.
2. Press **Enter**. The DO Barometric Pressure Entry Screen is displayed.

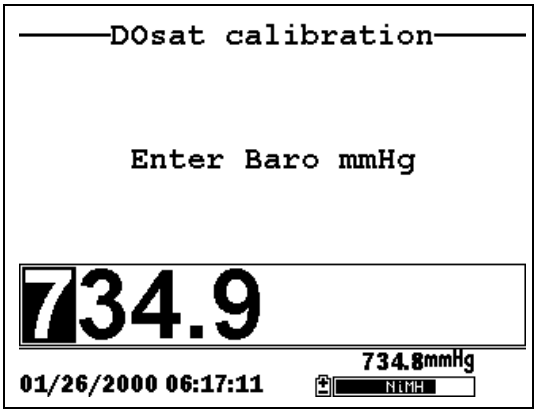


Figure 6.8 DO Barometric Pressure Entry Screen

3. Place approximately 3 mm (1/8 inch) of water in the bottom of the transport/calibration cup.

4. Place the probe module into the transport/calibration cup.

   NOTE: Make sure that the DO and temperature sensors are **not** immersed in the water.

5. Engage only 1 or 2 threads of the transport/calibration cup to ensure the DO sensor is vented to the atmosphere.

6. Use the keypad to enter the current local barometric pressure.

   NOTE: If the unit has the optional barometer, no entry is required.

   NOTE: Barometer readings that appear in meteorological reports are generally corrected to sea level and must be uncorrected before use (refer to Section *10.10 Calibrate Barometer, Step 2*).

7. Press **Enter**. The DO% saturation calibration screen is displayed.

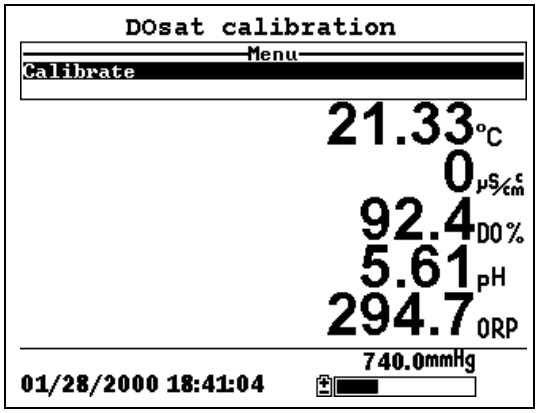


Figure 6.9 DO Sat Calibration Screen

8. Allow approximately ten minutes for the air in the transport/calibration cup to become water saturated and for

the temperature to equilibrate before proceeding. The current values of all enabled sensors will appear on the screen and will change with time as they stabilize.

9. Observe the reading under DO %. When the reading shows no significant change for approximately 30 seconds, press **Enter**. The screen will indicate that the calibration has been accepted and prompt you to press **Enter** again to Continue. See Figure 6.6 Calibrated.

10. Press **Enter**. This returns you to the DO calibration screen, See Figure 6.7 DO Calibration Screen.

11. Press **Escape** to return to the calibrate menu. See Figure 6.2 Calibrate Screen.

12. Rinse the probe module and sensors in tap or purified water and dry.

DO Calibration in mg/L

DO calibration in mg/L is carried out in a water sample which has a known concentration of dissolved oxygen (usually determined by a Winkler titration).

1. Go to the DO calibrate screen as described in Section *6.2.3 Dissolved Oxygen Calibration*, steps 1 through 3.

2. Use the arrow keys to highlight the **DO mg/L** selection.

3. Press **Enter**. The DO mg/L Entry Screen is displayed.

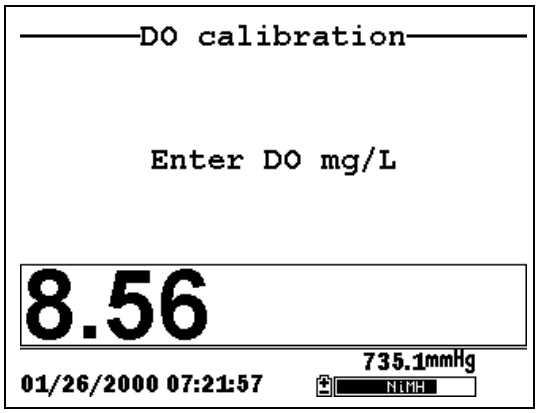


Figure 6.10 DO mg/L Entry Screen

4. Place the probe module in water with a known DO concentration.

   NOTE: Be sure to completely immerse all the sensors.

5. Use the keypad to enter the known DO concentration of the water.

6. Press **Enter**. The Dissolved Oxygen mg/L Calibration Screen is displayed.

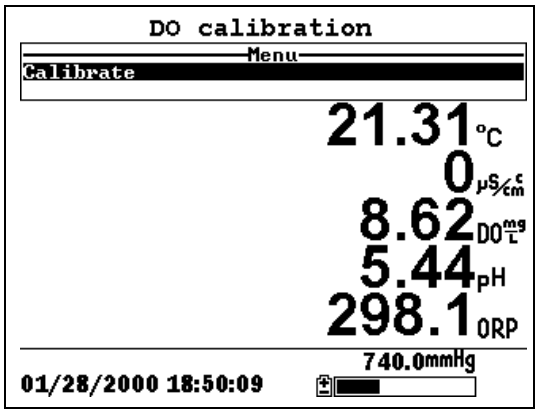


Figure 6.11 DO mg/L Calibration Screen

7. Stir the water with a stir bar, or by rapidly moving the probe module, to provide fresh sample to the DO sensor.

8. Allow at least one minute for temperature equilibration before proceeding. The current values of all enabled sensors will appear on the screen and will change with time as they stabilize.

9. Observe the DO mg/L reading, when the reading is stable (shows no significant change for approximately 30 seconds), press **Enter**. The screen will indicate that the calibration has been accepted and prompt you to press **Enter** again to Continue.

10. Press **Enter**. This returns you to the DO calibration screen. See Figure 6.7 DO Calibration Screen.

11. Press **Escape** to return to the calibrate menu. See Figure 6.2 Calibrate Screen.

12. Rinse the probe module and sensors in tap or purified water and dry.

6.2.4 pH Calibration

1. Go to the calibrate screen as described in *Section 6.2.1 Accessing the Calibrate Screen*.

2. Use the arrow keys to highlight the **pH** selection. See Figure 6.2 Calibrate Screen.

3. Press **Enter**. The pH calibration screen is displayed.

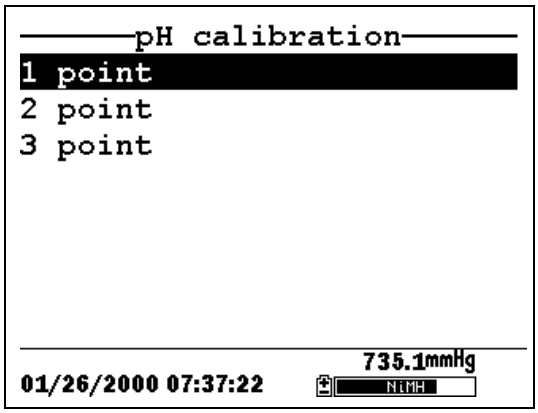


Figure 6.12 pH Calibration Screen

- Select the **1-point** option only if you are adjusting a previous calibration. If a 2-point or 3-point calibration has been performed previously, you can adjust the calibration by carrying out a one point calibration. The procedure for this calibration is the same as for a 2-point calibration, but the software will prompt you to select only one pH buffer.
- Select the **2-point** option to calibrate the pH sensor using only two calibration standards. Use this option if the media being monitored is known to be either basic or acidic. For example, if the pH of a pond is known to vary between 5.5 and 7, a two-point calibration with pH 7 and pH 4 buffers is sufficient. A three point calibration with an additional pH 10 buffer will not increase the accuracy of this measurement since the pH is not within this higher range.
- Select the **3-point** option to calibrate the pH sensor using three calibration solutions. In this procedure, the pH sensor is calibrated with a pH 7 buffer and two additional buffers. The 3-point calibration method assures maximum accuracy when the pH of the media to be monitored cannot be anticipated. The procedure for this calibration is the same as for a 2-point calibration, but the software will prompt you to select a third pH buffer.

4. Use the arrow keys to highlight the **2-point** selection.

5. Press **Enter**. The pH Entry Screen is displayed.

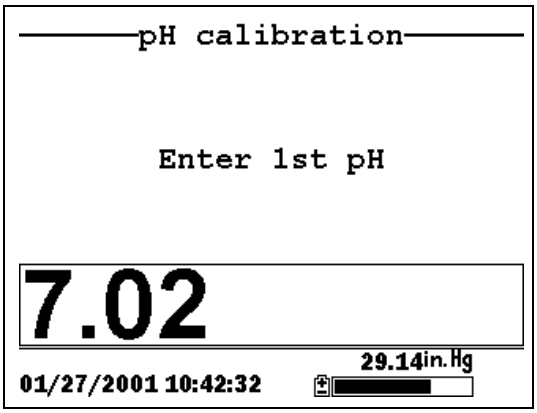


Figure 6.13 pH Entry Screen

6. Place the correct amount (see Table 6.1 Calibration Volumes) of pH buffer into a clean, dry or pre-rinsed transport/calibration cup.

⚠ **WARNING**: Calibration reagents may be hazardous to your health. See *Appendix D Health and Safety* for more information.

NOTE: For maximum accuracy, the pH buffers you choose should be within the same pH range as the water you are preparing to sample.

NOTE: Before proceeding, ensure that the sensor is as dry as possible. Ideally, rinse the pH sensor with a small amount of buffer that can be discarded. Be certain that you avoid cross-contamination of buffers with other solutions.

7. Carefully immerse the sensor end of the probe module into the solution.

8. Gently rotate and/or move the probe module up and down to remove any bubbles from the pH sensor.

NOTE: The sensor must be completely immersed. Using the recommended volumes from Table 6.1 Calibration Volumes, should ensure that the sensor is covered.

9. Screw the transport/calibration cup on the threaded end of the probe module and securely tighten.

   NOTE: Do not overtighten as this could cause damage to the threaded portions.

10. Use the keypad to enter the calibration value of the buffer you are using **at the current temperature**.

    NOTE: pH vs. temperature values are printed on the labels of all YSI pH buffers.

11. Press **Enter**. The pH calibration screen is displayed.

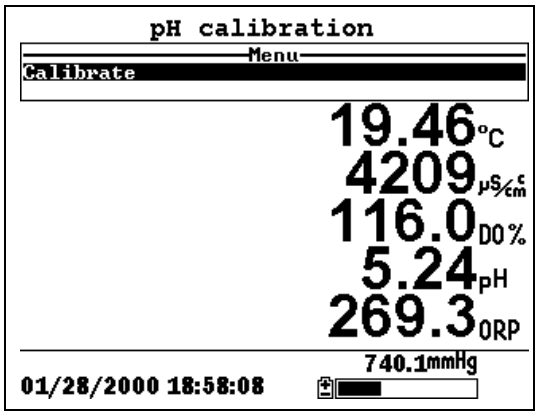


Figure 6.14 pH Calibration Screen

12. Allow at least one minute for temperature equilibration before proceeding. The current values of all enabled sensors will appear on the screen and will change with time as they stabilize.

13. Observe the reading under pH, when the reading shows no significant change for approximately 30 seconds, press **Enter**. The screen will indicate that the calibration has been accepted and prompt you to press **Enter** again to Continue.

14. Press **Enter**. This returns you to the Specified pH Calibration Screen, See Figure 6.13 pH Entry Screen.

15. Rinse the probe module, transport/calibration cup and sensors in tap or purified water and dry.

16. Repeat steps 6 through 13 above using a second pH buffer.

17. Press **Enter**. This returns you to the pH Calibration Screen, See Figure 6.12 pH Calibration Screen.

18. Press **Escape** to return to the calibrate menu. See Figure 6.2 Calibrate Screen.

19. Rinse the probe module and sensors in tap or purified water and dry.

6.2.5 ORP Calibration

1. Go to the calibrate screen as described in Section *6.2.1 Accessing the Calibrate Screen*.

2. Use the arrow keys to highlight the **ORP** selection. See Figure 6.2 Calibrate Screen.

3. Press **Enter**. The ORP calibration screen is displayed.

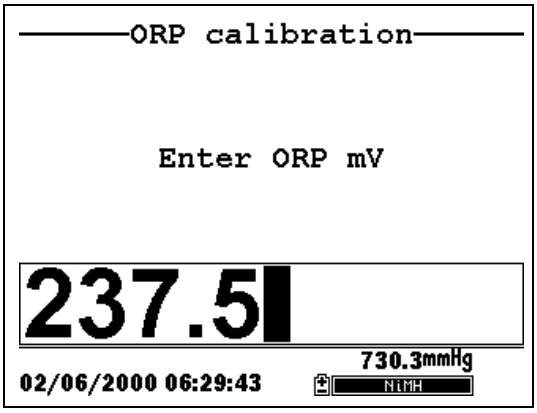


Figure 6.15 Specified ORP Calibration Screen

4. Place the correct amount (see Table 6.1 Calibration Volumes) of a known ORP solution (we recommend Zobell solution) into a clean, dry or pre-rinsed transport/calibration cup.

⚠ **WARNING**: Calibration reagents may be hazardous to your health. See *Appendix D Health and Safety* for more information.

NOTE: Before proceeding, ensure that the sensor is as dry as possible. Ideally, rinse the ORP sensor with a small amount of solution that can be discarded. Be certain that you avoid cross-contamination with other solutions.

5. Carefully immerse the sensor end of the probe module into the solution.

6. Gently rotate and/or move the probe module up and down to remove any bubbles from the ORP sensor.

   NOTE: The sensor must be completely immersed. Using the recommended volumes from Table 6.1 Calibration Volumes should ensure that the sensor is covered.

7. Screw the transport/calibration cup on the threaded end of the probe module and securely tighten.

   NOTE: Do not overtighten as this could cause damage to the threaded portions.

8. Use the keypad to enter the correct value of the calibration solution you are using at the current temperature. Refer to Table 6.2 Zobel Solution Values.

Temperature °C	Zobell Solution Value, mV
-5	270.0
0	263.5
5	257.0
10	250.5
15	244.0
20	237.5
25	231.0
30	224.5
35	218.0
40	211.5
45	205.0
50	198.5

Table 6.2 Zobel Solution Values

9. Press **Enter**. The ORP calibration screen is displayed.

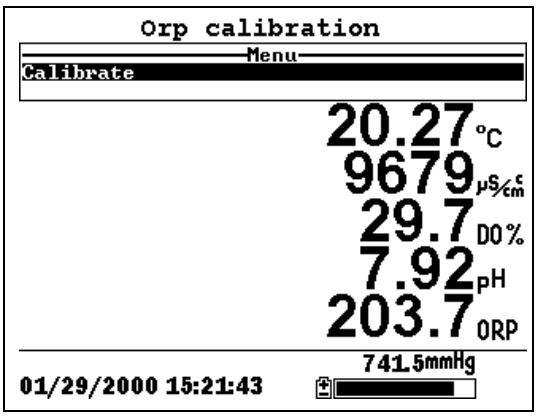


Figure 6.16 ORP Calibration Screen

10. Allow at least one minute for temperature equilibration before proceeding. The current values of all enabled sensors will appear on the screen and will change with time as they stabilize.

NOTE: Verify that the temperature reading matches the value you used in Table 6.2 Zobel Solution Values.

11. Observe the reading under ORP, when the reading shows no significant change for approximately 30 seconds, press **Enter**. The screen will indicate that the calibration has been accepted and prompt you to press **Enter** again to Continue.

12. Press **Enter**. This returns you to the Calibrate Screen. See Figure 6.2 Calibrate Screen.

13. Rinse the probe module and sensors in tap or purified water and dry.

6.3 Return to Factory Settings

1. Go to the calibrate screen as described in Section *6.2.1 Accessing the Calibrate Screen*.

2. Use the arrow keys to highlight the **Conductivity** selection. See Figure 6.2 Calibrate Screen.

   NOTE: We will use the Conductivity sensor as an example; however, this process will work for any sensor.

3. Press **Enter.** The Conductivity Calibration Selection Screen is displayed. See Figure 6.3 Conductivity Calibration Selection Screen.

4. Use the arrow keys to highlight the **Specific Conductance** selection.

5. Press **Enter.** The Conductivity Calibration Entry Screen is displayed. See Figure 6.4 Conductivity Calibration Entry Screen.

6. Press and hold the **Enter** key down and press the **Escape** key.

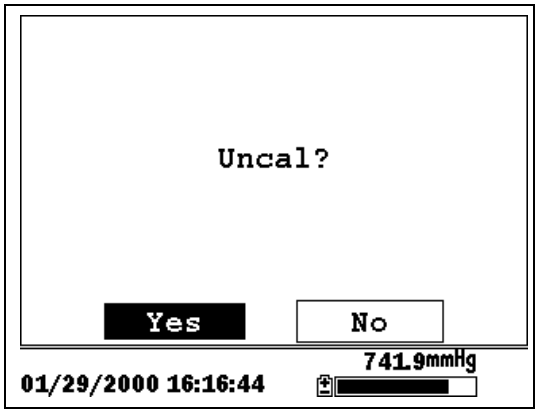


Figure 6.17 ORP Calibration Screen

7. Use the arrow keys to highlight the **YES** selection.

CAUTION: This returns a sensor to the factory settings. For example, in selecting to return specific conductance to the factory setting, salinity and conductivity will automatically return to their factory settings.

8. Press **Enter**. This returns you to the Conductivity Calibrate Selection Screen, See Figure 6.3 Conductivity Calibration Selection Screen.

9. Press **Escape** to return to the calibrate menu. See Figure 6.2 Calibrate Screen.

7. Run

The Run screen displays data from the sensors in real-time and allows the user to log sample data to memory for later analysis. Refer to Section *9 Logging* for details on logging sample data.

7.1 Real-Time Data

NOTE: Before measuring samples you must prepare the probe module (refer to Section *3.4 Preparing the Probe Module*), attach the probe module to the instrument (refer to Section *3.6 Instrument/Cable Connection*) and calibrate the sensors (refer to Section *6 Calibrate*).

1. Press the **On/off** key.

OR select Run from the main menu to display the run screen.

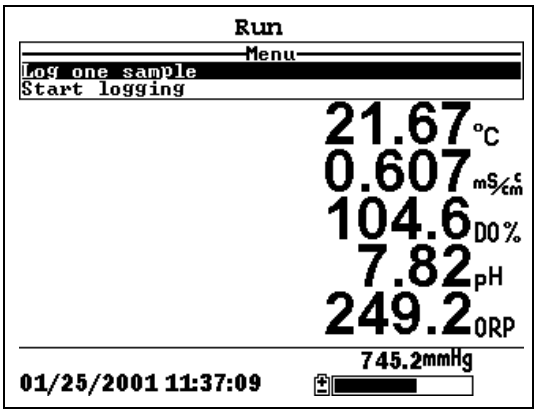


Figure 7.1 Run Screen

2. Make sure the probe sensor guard is installed.

3. Place the probe module in the sample. Be sure to completely immerse all the sensors.

4. Rapidly move the probe module through the sample to provide fresh sample to the DO sensor.

5. Watch the readings on the display until they are stable.

6. Refer to Section *9 Logging* for instructions on logging sample data.

8. File

The File menu allows the user to view, upload or delete sample data and calibration record files stored in the YSI 556 MPS.

8.1 Accessing the File Screen

1. Press the **On/off** key to display the run screen.

2. Press the **Escape** key to display the main menu screen.

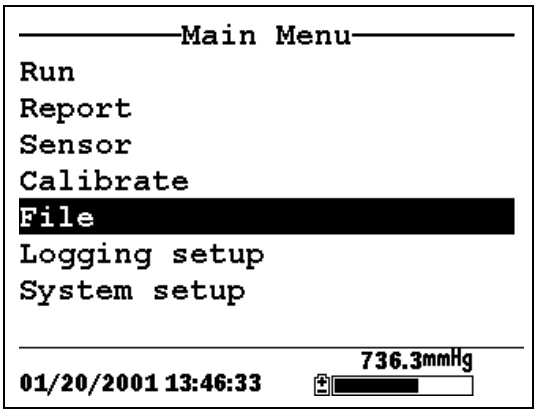


Figure 8.1 Main Menu Screen

3. Use the arrow keys to highlight the **File** selection.

4. Press the **Enter** key. The file screen is displayed.

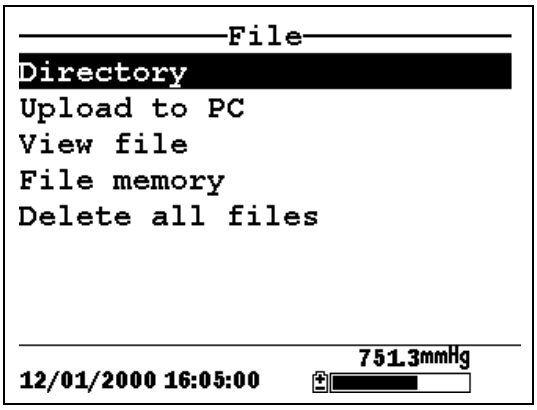


Figure 8.2 File Screen

8.2 Directory

1. Go to the file screen as described in Section *8.1 Accessing the File Screen*.

2. Use the arrow keys to highlight the **Directory** selection. See Figure 8.2 File Screen.

3. Press the **Enter** key. The file list screen is displayed.

   NOTE: Files are listed in the order in which they are logged to memory. Sample Data files have the file extension **.dat**, while Calibration Record files have the file extension **.glp**.

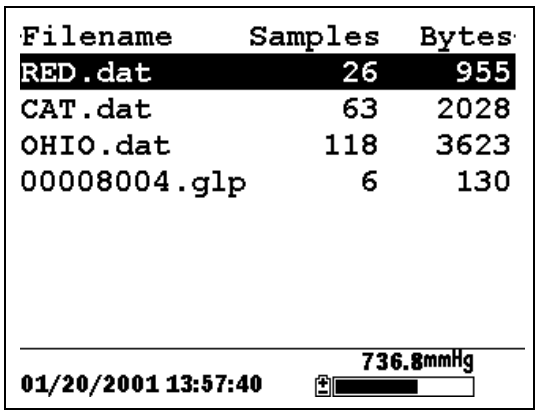


Figure 8.3 File List Screen

4. Use the arrow keys to highlight a file.

5. Press the **Enter** key. The file details screen is displayed.

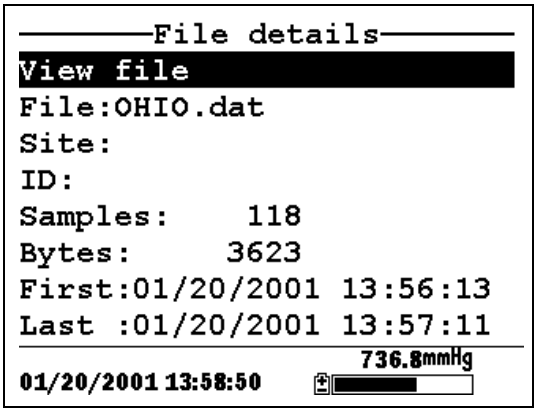


Figure 8.4 File Details Screen

6. Press the **Enter** key to view the file data. Refer to Section *8.3 View File* for details.

7. Press the **Escape** key repeatedly to return to the main menu screen.

8.3 View File

1. Go to the file screen as described in Section *8.1 Accessing the File Screen*. See Figure 8.2 File Screen.

2. Use the arrow keys to highlight the **View file** selection.

3. Press the **Enter** key. A list of files is displayed. See Figure 8.3 File List Screen.

4. Use the arrow keys to highlight an individual file.

   NOTE: You may have to scroll down to see all the files.

5. Press the **Enter** key. The file data is displayed with the file name at the top of the display.

   NOTE: If no file name was specified, the data is stored under the default name NONAME1.dat.

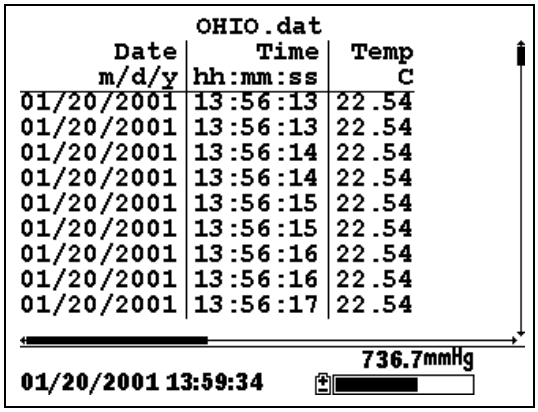


Figure 8.5 File Data Screen

6. Use the arrow keys to scroll horizontally and/or vertically to view all the data.

7. Press the **Escape** key repeatedly to return to the main menu screen.

8.4 Upload to PC

EcoWatch™ for Windows™ must be used as the PC software interface to the YSI 556 MPS. Refer to *Appendix G EcoWatch* for more information. EcoWatch for Windows is available at no cost via a download from the YSI Web Site (www.ysi.com) or by contacting YSI Customer Support. Refer to *Appendix E Customer Service.*

8.4.1 Upload Setup

1. Disconnect the YSI 5563 Probe Module from the YSI 556 MPS instrument.

2. Connect the YSI 556 MPS to a serial (Comm) port of your computer via the 655173 PC Interface cable as shown in the following diagram:

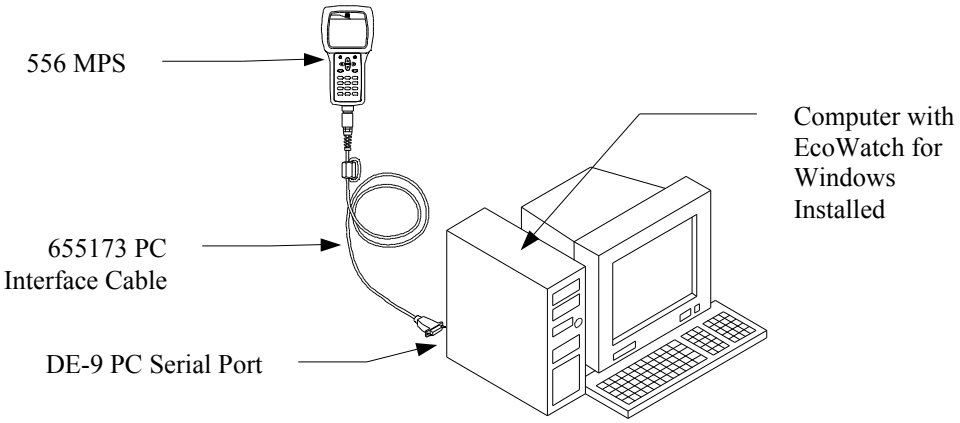


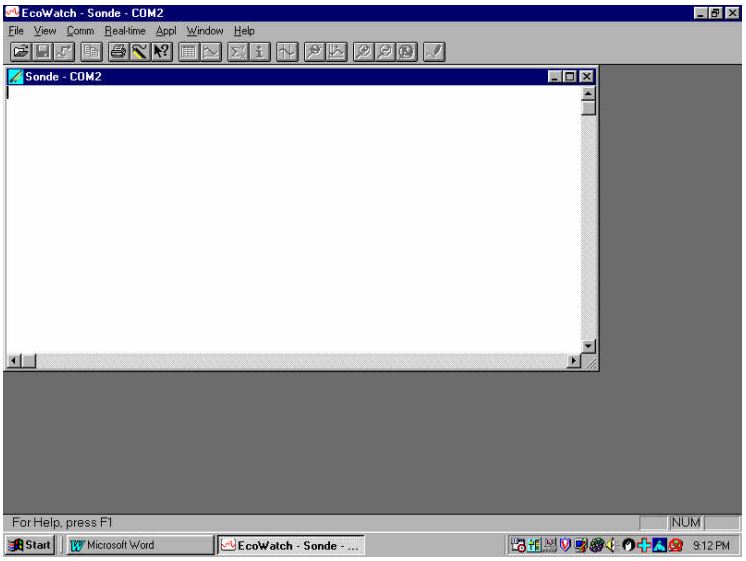
Figure 8.6 Computer/Instrument Interface

3. Open EcoWatch for Windows on your computer.

NOTE: See *Appendix G EcoWatch* for installation instructions.

4. Click on the sonde/probe icon in the upper toolbar.

5. Set the Comm port number to match the port the YSI 556 MPS is connected to. After this setup procedure, the following screen will be present on your PC monitor:



8.4.2 Uploading a .DAT File

1. Setup the instrument as described in Section *8.4.1 Upload Setup*.

2. Go to the YSI 556 MPS file screen as described in Section *8.1 Accessing the File Screen.*

3. Use the arrow keys to highlight the **Upload to PC** selection. See Figure 8.2 File Screen.

4. Press the **Enter** key. The file list screen is displayed. See Figure 8.3 File List Screen.

5. Use the arrow keys to highlight the DAT file that you wish to transfer and press **Enter**, both the YSI 556 MPS and PC displays show the progress of the file transfer.

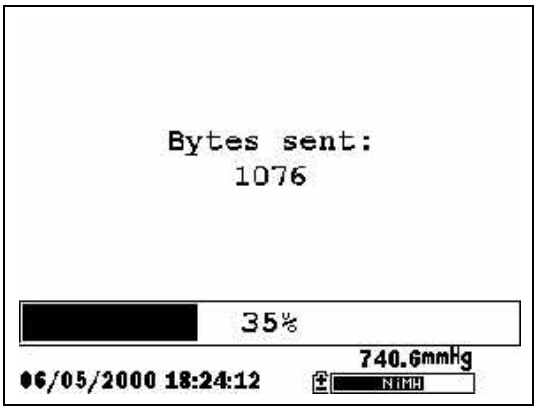
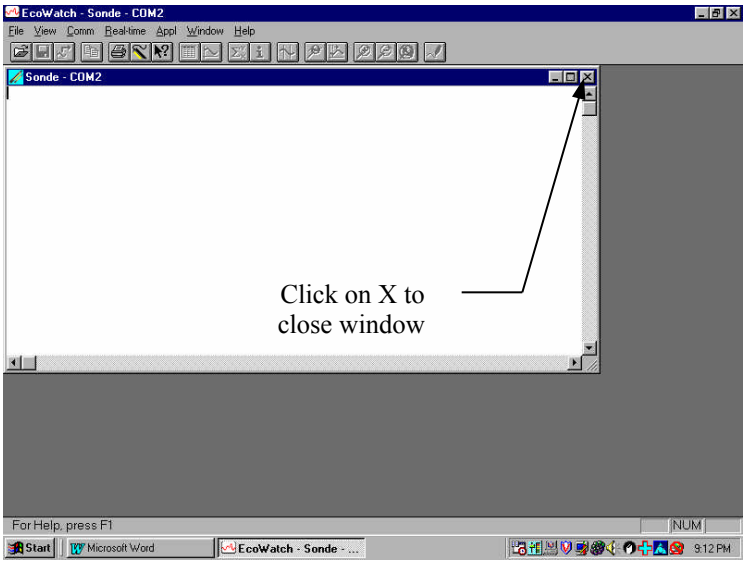


Figure 8.7 File Transfer Progress Screen

NOTE: After transfer, the file will be located in the C:\ECOWWIN\DATA folder of your PC, designated with a .DAT extension.

6. After the file transfer is complete, close the terminal window (small window on the PC) by clicking on the "X" at its upper right corner.



7. Press the **Escape** key on the YSI 556 MPS repeatedly to return to the main menu screen.

8.4.3 Uploading a Calibration Record (.glp) File

For more information on the calibration record, Refer to *Appendix H Calibration Record Information.*

1. Setup up the instrument as described in Section *8.4.1 Upload Setup*.

2. Go to the YSI 556 MPS file screen as described in Section *8.1 Accessing the File Screen.*

3. Use the arrow keys to highlight the **Upload to PC** selection. See Figure 8.2 File Screen.

4. Press the **Enter** key. The file list screen is displayed. See Figure 8.3 File List Screen.

5. Use the arrow keys to highlight the calibration record file that you wish to transfer and press **Enter.**

6. You will then be given a choice of uploading the file in three formats; **Binary, Comma & “” Delimited, and ASCII Text**.

   NOTE: The binary format is reserved for future YSI software packages.

7. Choose an option and press **Enter,** both the YSI 556 and PC displays show the progress of the file transfer.

   NOTE: After transfer, the file will be located in the C:\ECOWWIN\DATA folder of your PC, designated with the appropriate file extension.

   NOTE: To view the Calibration Record data after upload, simply open the .txt file in a general text editor such as Wordpad or Notepad.

8. After the file transfer is complete, close the terminal window (small window on the PC) by clicking on the “X” at its upper right corner.

9. Press the **Escape** key repeatedly to return to the main menu screen.

8.5 File Memory

1. Go to the file screen as described in Section *8.1 Accessing the File Screen*.

2. Use the arrow keys to highlight the **File memory** selection. See Figure 8.2 File Screen.

3. Press the **Enter** key. The file bytes used screen is displayed.

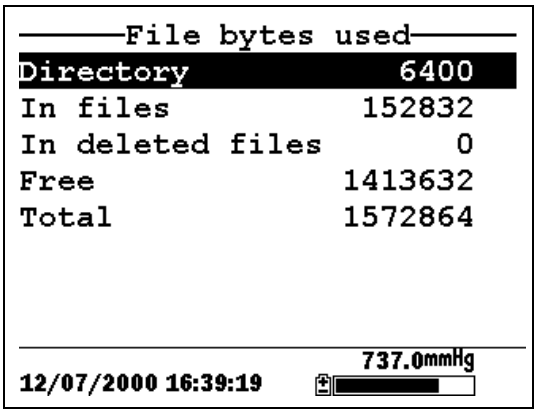


Figure 8.8 File Bytes Used Screen

4. The amount of free memory is listed in line 4 of the file bytes used screen.

   NOTE: If the amount of free memory is low, it may be time to delete all files (after first uploading all data to a PC). Refer to Section *8.6 Delete All Files*.

5. Press the **Escape** key repeatedly to return to the main menu screen.

8.6 Delete All Files

NOTE: It is not possible to delete individual files in order to free up memory. The only way to free up memory is to delete ALL files present. Take care to transfer all files to your computer (refer to Section *8.4 Upload to PC*) *before* deleting them.

1. Go to the file screen as described in Section *8.1 Accessing the File Screen*.

2. Use the arrow keys to highlight the **Delete all files** selection. See Figure 8.2 File Screen.

3. Press the **Enter** key. The Delete all Files screen is displayed.

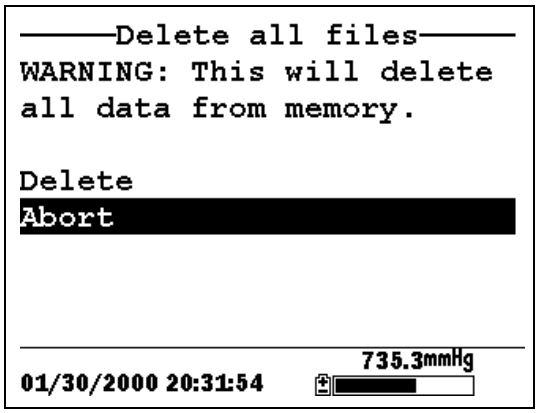


Figure 8.9 Delete All Files Screen

4. Use the arrow keys to highlight the **Delete** selection.

5. Press the **Enter** key.

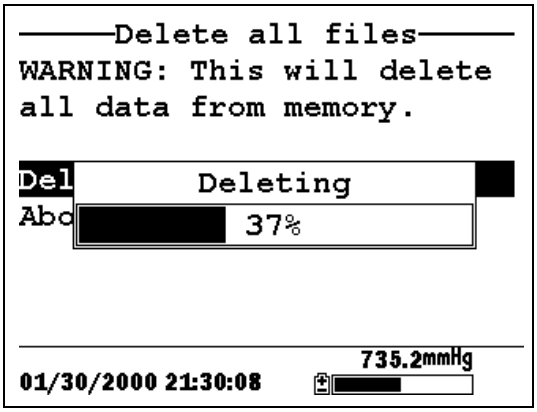


Figure 8.10 Deleting

The progress of file deletion is displayed in bar graph format.

NOTE: Deleting all files in the directory will not change any information in the site list.

6. Press the **Escape** key repeatedly to return to the main menu screen.

9. Logging

9.1 Accessing the Logging Setup Screen

1. Press the **On/off** key to display the run screen.

2. Press the **Escape** key to display the main menu screen.

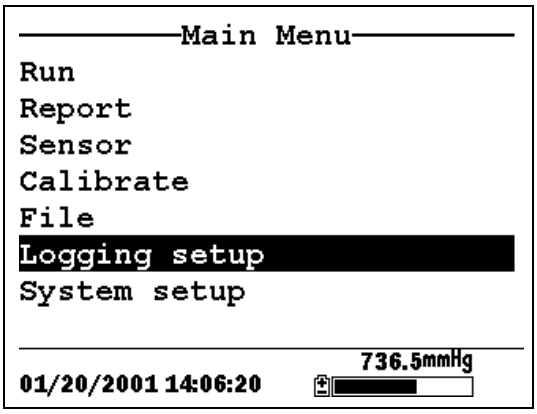


Figure 9.1 Main Menu

3. Use the arrow keys to highlight the **Logging setup** selection.

4. Press the **Enter** key. The logging setup screen is displayed.

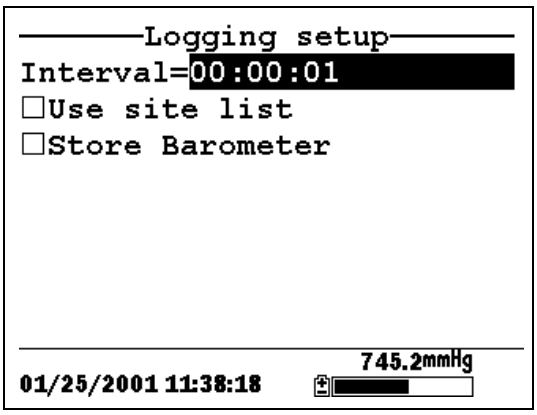


Figure 9.2 Logging Setup Screen

9.2 Setting Logging Interval

Follow steps below to set the interval for logging a data stream.

NOTE: If you do not specify an interval, the instrument will use a default interval setting of 1 second.

NOTE: It is not necessary to set a logging interval when logging a single sample.

1. Go to the logging setup screen as described in Section *9.1 Accessing the Logging Setup Screen*.

2. Use the keypad to enter an interval between 1 second and 15 minutes. Refer to Section *2.9 Keypad Use*.

   NOTE: The interval field has hour, minute and second entry fields. Any entry over 15 minutes will change automatically to a 15-minute setting.

3. Press the **Enter** key. The data stream interval is set.

4. Press the **Escape** key repeatedly to return to the main menu screen.

9.3 Storing Barometer Readings

NOTE: The **Store barometer** option is only available on instruments that are equipped with the optional barometer.

1. Go to the logging setup screen as described in Section *9.1 Accessing the Logging Setup Screen*.

2. Use the arrow keys to highlight the **Store barometer** selection. See Figure 9.2 Logging Setup Screen.

3. Press the **Enter** key until a check mark is entered in the box next to the store barometer selection if you want to log barometric readings.

   OR press the **Enter** key until the box next to the barometer selection is empty if you do not want to log barometric readings.

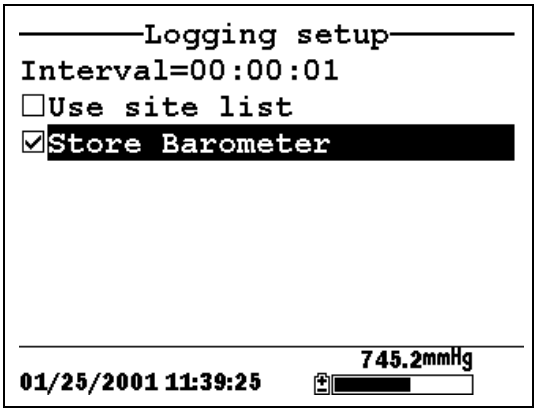


Figure 9.3 Store Barometer

4. Press the **Escape** key repeatedly to return to the main menu screen.

9.4 Creating a Site List

The site list option allows you to define file and site descriptions in the office or laboratory before moving to field logging studies. This is usually more convenient than entering the information at the site and is particularly valuable if you are visiting certain sites on a regular basis. The following section describes how to set up site lists which contain entries designated "Site Descriptions" that will be instantly available to the user in the field to facilitate the logging of data with pre-established naming of files and sites. There are two kinds of **Site Descriptions** available for use in Site lists:

- **Site Descriptions** associated with applications where data from a single site is always logged to a single file. This type is referred to as a "Single-Site Description" and is characterized by two parameters – a file name and a site name. Files logged to YSI 556 MPS memory under a **Single-Site Description** will be characterized primarily by the file name, but will also have the Site name attached, so that it is viewable in either the YSI 556 MPS **File directory** or in EcoWatch for Windows after upload to a PC
- **Site Descriptions** associated with applications where data from multiple sites are logged to a single file. This type is

referred to as a “Multi-site Description” and is characterized by three parameters – a file name, a site name, and a site number. Files logged to YSI 556 MPS memory under a **Multi-site Description** are characterized by a file name, but not a site name, since multiple sites are involved. However, each data point has a Site Number attached to it so that the user can easily determine the sampling site when viewing the data from the YSI 556 MPS **File** menu or processing the data in EcoWatch for Windows after upload to a PC.

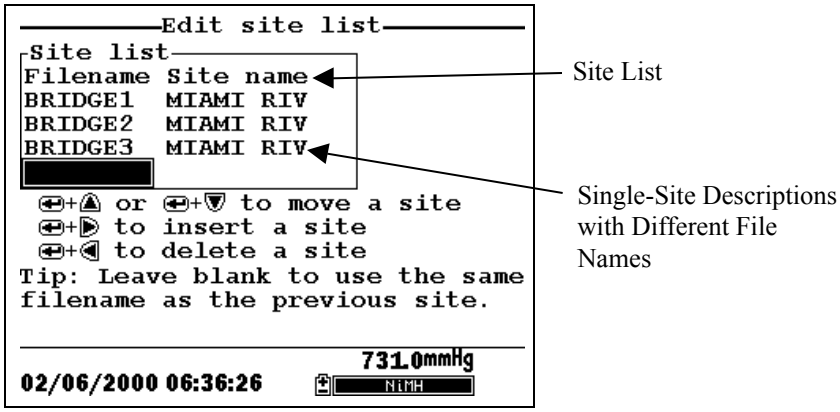


Figure 9.4 Single-Site Descriptions

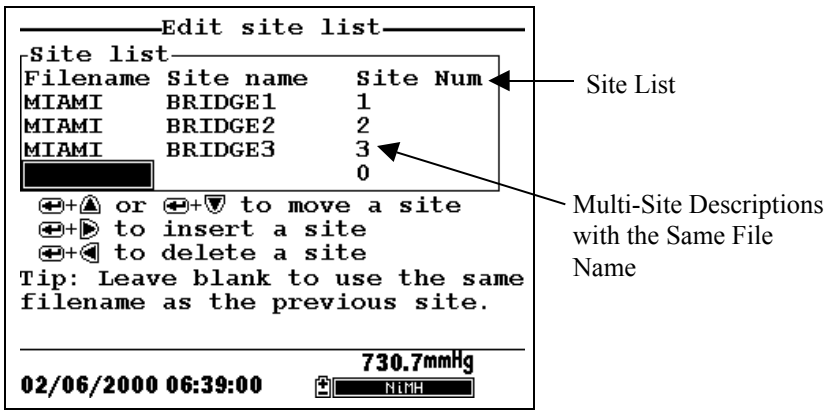


Figure 9.5 Multiple-Site Descriptions

NOTE: Site lists containing Single Site Descriptions are usually input with the designation **Store Site Number** INACTIVE in the YSI 556 MPS **Logging setup** menu. Thus, no site numbers

appear in the first **Site list** example. Conversely, **Site lists** containing **Multi-Site Descriptions** MUST be input with the **Store Site Number** selection ACTIVE as shown in the second example.

To create a site list:

1. Go to the logging setup screen as described in Section *9.1 Accessing the Logging Setup Screen*.

2. Use the arrow keys to highlight the **Use site list** selection.

3. Press the **Enter** key. A check mark is entered in the box next to the use site list selection *and* two new entries appear on the logging setup screen. See Figure 9.6 Logging Setup Screen.

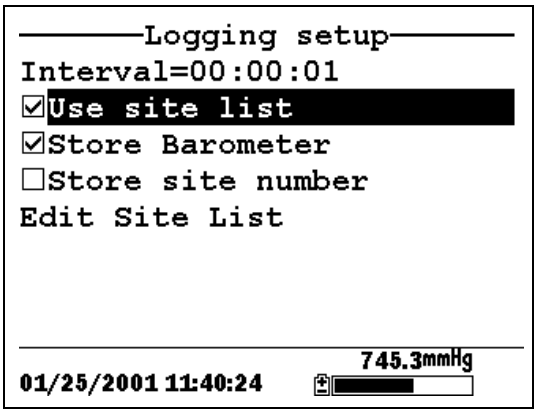


Figure 9.6 Logging Setup Screen

4. Use the arrow keys to highlight the **Store site number** selection.

5. If you are creating Multi-Site Descriptions (which require that the site **number** be stored in your data files), press the **Enter** key until a check mark appears in the box next to the store site number selection.

   OR Press the **Enter** key until the box next to the store site number selection is empty, to create Single-Site

Descriptions. The site **name** will be stored in the header of your data files.

6. Use the arrow keys to highlight the **Edit site list** selection.

7. Press the **Enter** key. The edit site list screen is displayed. See Figure 9.7 Edit Site List Screen. The **Filename** field is ready for input.

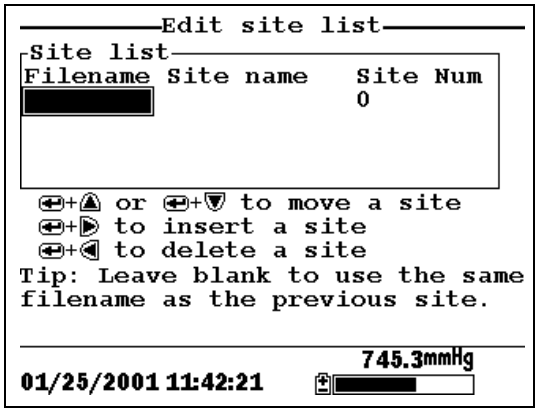


Figure 9.7 Edit Site List Screen

8. Use the keypad to enter a filename up to 8 characters in length. Refer to Section *2.9 Keypad Use*.

9. Press the **Enter** key. The cursor moves to the right for the entry of a **Site name**.

10. Use the keypad to enter a site name up to 11 characters in length. Refer to Section *2.9 Keypad Use*.

    NOTE: If the store site number selection is *not* checked, skip to Step 13.

11. Press the **Enter** key. The cursor moves to the site number entry position.

12. Use the keypad to enter a site number up to 7 characters in length. Refer to Section *2.9 Keypad Use*.

13. Press **Enter**. The cursor moves to the next filename entry position.

14. Repeat Steps 8 to 13 until all filenames and sites have been entered.

15. Press **Escape** repeatedly to return to the main menu screen.

9.5 Editing a Site List

1. Go to the logging setup screen as described in Section *9.1 Accessing the Logging Setup Screen*.

2. Use the arrow keys to highlight the **Edit Site List** selection. See Figure 9.6 Logging Setup Screen.

3. Press the **Enter** key. The edit site list screen is displayed.

4. Edit the site list using the keystrokes described below.

   NOTE: Editing the site list will not have any effect on files stored in the instrument memory.

To MOVE a site: **Use the arrow keys to highlight a site. Press the Up or Down arrow key while holding down the Enter key.**

To INSERT a site above another site: **Use the arrow keys to highlight the site. Press the** Right **arrow key while holding down the** Enter **key. Use keypad to input letters. Refer to Section *2.9 Keypad Use*.**

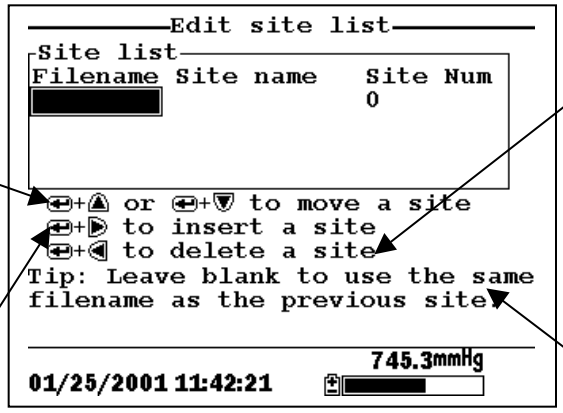


Figure 9.8 Keystrokes for Editing Site List

To DELETE a site: **Use the arrow keys to highlight a site. Press the Left arrow key while holding down the Enter key.**

To use the same file name as the previous site: **Leave the filename blank.**

9.6 Logging Data Without a Site List

1. Follow Steps 1 through 5 in Section 7.1 Real-Time Data.

2. Use the arrow keys to highlight the **Log one sample** selection on the run screen if only a single sample is being logged.

   OR Use the arrow keys to highlight the **Start logging** selection on the run screen if a data stream is being logged.

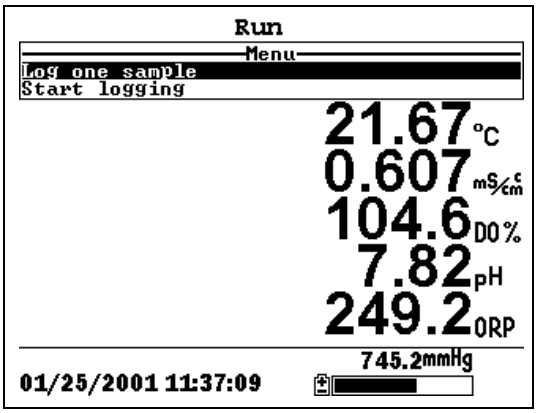


Figure 9.9 Run Screen

3. Press the **Enter** key. The Enter information screen is displayed.

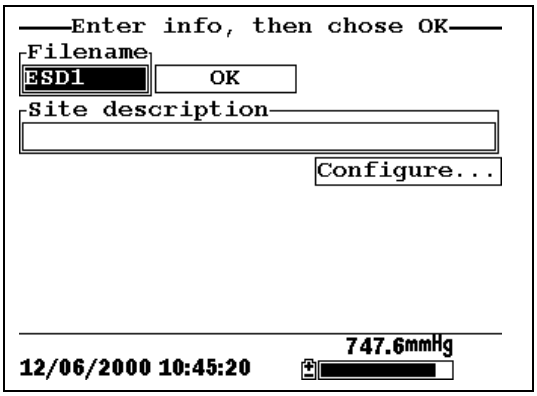


Figure 9.10 Enter Information Screen

NOTE: The last filename used will be displayed.

4. Use the keypad to enter a file name. Refer to Section *2.9 Keypad Use*.

   NOTE: The instrument will assign a default file name of NONAME if no file name is specified.

5. Press the **Enter** key to input the file name.

6. Use the arrow keys to highlight the **Site description** field in the enter information screen.

   NOTE: Entering a Site Description is optional. You may leave the Site Description blank and skip to Step 9.

7. Use the keypad to enter a site description name. Refer to Section *2.9 Keypad Use*.

8. Press the **Enter** key to input the site description.

   NOTE: If you want to change the logging setup, such as sampling interval or storing the barometer reading, use the arrow keys to highlight the **Configure** field, press the **Enter** key, then refer to Section *9.2 Setting Logging Interval* or *9.3 Storing Barometer Readings* for details.

9. Use the arrow keys to highlight the **OK** field in the center of the information screen.

10. Press the **Enter** key to start logging.

    NOTE: If the parameter mismatch screen is displayed, refer to Section *9.8 Adding Data to Existing Files*.

11. If a single point is being logged, the header on the run screen changes momentarily from **Menu** to **Sample logged** to confirm that the point was successfully logged. Skip to Step 13.

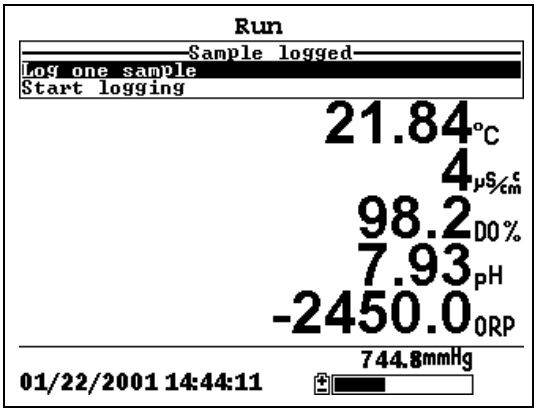


Figure 9.11 Sample Logged Screen

If a continuous stream of points is being logged, the start logging entry in the run screen changes from **Start logging** to **Stop logging**.

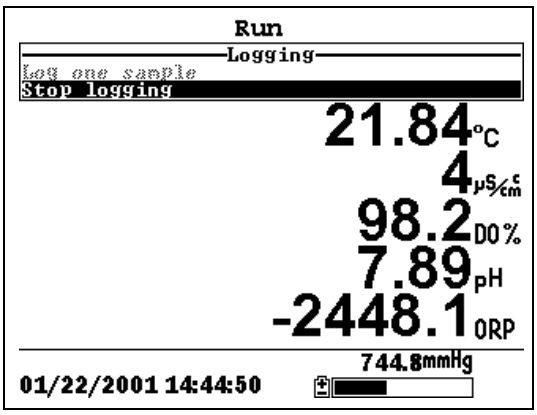


Figure 9.12 Logging Screen

12. At the end of the logging interval, press **Enter** to stop logging.

13. Refer to Section *8.3 View File* to view the data on the instrument display.

9.7 Logging Data With a Site List

1. If you have not already created a site list, refer to Section *9.4 Creating a Site List*.

2. Follow Steps 1 through 5 in Section 7.1 Real-Time Data.

3. Use the arrow keys to highlight the **Log one sample** selection on the run screen if only a single sample is being logged.

   OR Use the arrow keys to highlight the **Start logging** selection on the run screen if a data stream is being logged. See Figure 9.9 Run Screen.

4. Press the **Enter** key. The Pick a site screen is displayed.

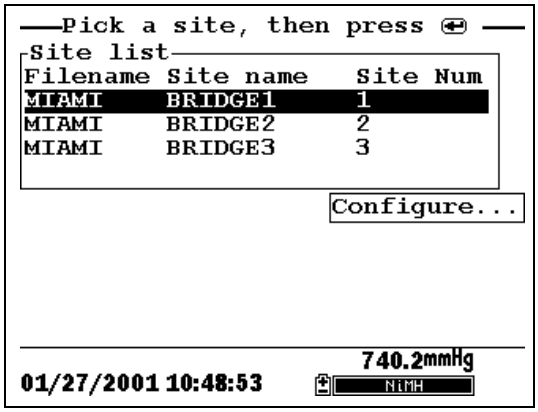


Figure 9.13 Pick a Site Screen

5. Use the arrow keys to highlight the **site** of your choice.

   NOTE: If the site of your choice is grayed out in the site list, refer to Section *9.8 Adding Data to Existing Files*.

   NOTE: Refer to Section *9.5 Editing a Site List* if you want to edit the site list.

6. Press the **Enter** key to start logging.

NOTE: If the parameter mismatch screen is displayed, refer to Section *9.8 Adding Data to Existing Files*.

7. If a single point is being logged, the header on the run screen changes momentarily from **Menu** to **Sample logged** to confirm that the point was successfully logged. See Figure 9.11 Sample Logged Screen. Skip to Step 9.

   If a continuous stream of points is being logged, the start logging entry in the run screen changes from **Start logging** to **Stop logging**. See Figure 9.12 Logging Screen.

8. At the end of the logging interval, press **Enter** to stop logging.

9. Refer to Section *8.3 View File* to view the data on the instrument display.

9.8 Adding Data to Existing Files

In order to add new data to an existing file, the current logging and sensor setup must be *exactly* the same as when the file was created. The following settings must be the same:

- **Sensors enabled** (refer to Section *4 Sensors*)
- **Store Barometer** (refer to Section *9.3 Storing Barometer Readings*)
- **Store Site Number** (refer to Section *9.4 Creating a Site List*)

If the current logging setup is not exactly the same as when the file was created, a parameter mismatch screen is displayed.

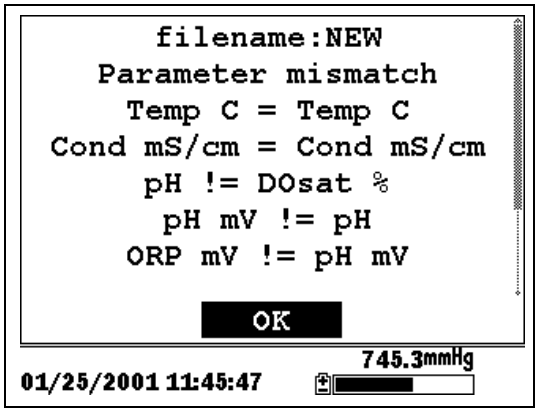


Figure 9.14 Parameter Mismatch Screen

NOTE: The right column shows parameters used when the file was created. The left column shows current parameters.

1. Press the **Down Arrow** key to scroll down and find the mismatch(es).

2. Use the following chart to resolve the mismatch(es).

Mismatch	Action	Reference
Sensor(s) missing from left column	Enable the missing sensor(s)	Section *4 Sensors*
Extra sensor(s) listed in left column	Disable the extra sensor(s)	Section *4 Sensors*
Barometer missing from left column, but present in right column	Enable the Store Barometer setting	Section *9.3 Storing Barometer Readings*
Barometer present in left column, but missing from right column	Disable the Store Barometer setting	Section *9.3 Storing Barometer Readings*
Store Site Number missing from left column, but present in right column	Enable the Store Site Number setting	Section *9.4 Creating a Site List*
Store Site Number present in left column, but missing from right column	Disable the Store Site Number setting	Section *9.4 Creating a Site List*

3. Return to Section *9.6 Logging Data Without a Site List* or *9.7 Logging Data With a Site List*.

10.System Setup

The YSI 556 MPS has a number of features that are user-selectable or can be configured to meet the user's preferences. Most of these choices are found in the **System setup** menu.

10.1 Accessing the System Setup Screen

1. Press the **On/off** key to display the run screen. See Figure 2.1 Front View of YSI 556 MPS.

2. Press the **Escape** key to display the main menu screen.

3. Use the arrow keys to highlight the **System setup** selection.

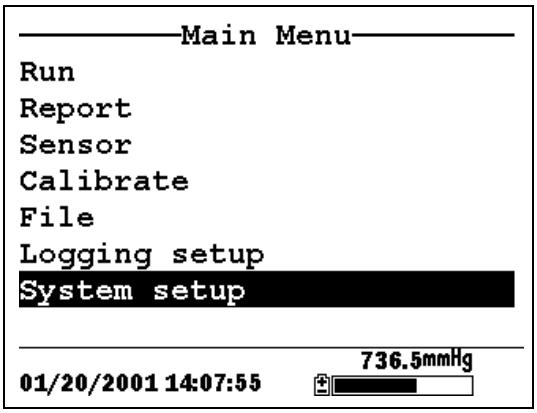


Figure 10.1 Main Menu

4. Press the **Enter** key. The system setup screen is displayed.

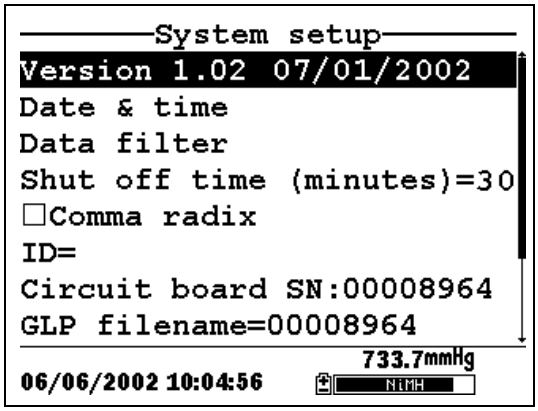


Figure 10.2 System Setup Screen

NOTE: The first line of the **System setup** menu shows the current software version of your YSI 556 MPS. As software enhancements are introduced, you will be able to upgrade your YSI 556 MPS from the YSI Web site. Refer to Section *11.2 Upgrading YSI 556 MPS* Software for details.

10.2 Date and Time Setup

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

2. Use the arrow keys to highlight the **Date & time** selection on the system setup screen. See Figure 10.2 System Setup Screen.

3. Press **Enter**. The date and time setup screen is displayed.

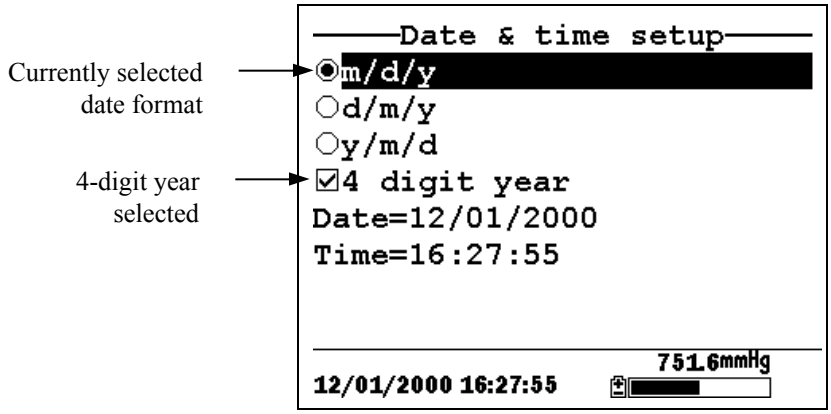


Figure 10.3 Date Setup Screen

NOTE: A black dot to the left of a date format indicates that format is selected.

4. Use the arrow keys to highlight your desired date format.

5. Press **Enter**.

6. Use the arrow keys to highlight the 4-digit year selection.

7. Press **Enter**. A check mark appears in the check box next to the 4-digit year selection.

   NOTE: If unchecked, a 2-digit year is used.

8. Use the arrow keys to highlight the **Date** selection.

9. Press **Enter**. A cursor appears over the first number in the date.

10. Enter the proper number from the keypad for the highlighted date digit. The cursor moves automatically to the next date digit. Refer to Section *2.9 Keypad Use* for more keypad information.

11. Repeat Step 10 until all date digits are correct.

12. Press **Enter** to input the specified date.

13. Use the arrow keys to highlight the **Time** selection.

14. Press **Enter**. A cursor appears over the first number in the time selection.

15. Enter the proper number from the keypad for the highlighted time digit. The cursor moves automatically to the next time digit.

    NOTE: Use military format when entering time. For example, 2:00 PM is entered as 14:00.

16. Repeat Step 15 until all time digits are correct.

17. Press **Enter** to input the correct time.

18. Press the **Escape** key repeatedly to return to the Main menu screen.

10.3 Data Filter

The Data Filter is a software filter that eliminates sensor noise and provides more stable readings.

NOTE: YSI recommends using the default values for the data filter for most field applications.

However, users who are primarily interested in a fast response from their dissolved oxygen sensor should consider a change of the default time constant setting of 8 seconds to one of 2 seconds. This change can be made according to the instructions in Section *10.3.1 Changing the Data Filter Settings* below. The disadvantage of lowering the time constant is that field pH readings may appear somewhat noisy if the cable is in motion.

10.3.1 Changing the Data Filter Settings

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

2. Use the arrow keys to highlight the **Data filter** selection. See Figure 10.1 Main Menu.

3. Press the **Enter** key. The Data filter setup screen is displayed.

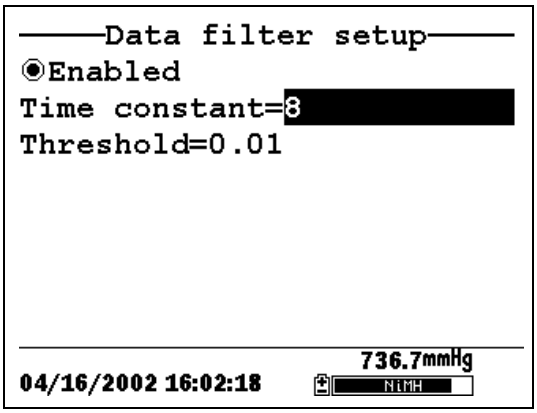


Figure 10.4 Data Filter Screen

4. With Enabled highlighted, press the **Enter** key to Enable or Disable the data filter. A black dot to the left of the selection indicates the data filter is enabled.

5. Use the arrow keys to highlight the **Time constant** field.

   NOTE: This value is the time constant in seconds for the software data filter. Increasing the time constant will result in greater filtering of the data, but will also slow down the apparent response of the sensors.

6. Use the keypad to enter a value. The default value is 8 and this value is ideal for most 556 field applications. As described in Section *10.3 Data Filter* above, users who wish to decrease the response time of the DO readings at the expense of some noise for the pH readings determined concurrently, should change the Time Constant to a value of 2.

7. Press the **Enter** key to enter the time constant.

8. Use the arrow keys to highlight the **Threshold** field.

   NOTE: This value determines when the software data filter will engage/disengage, speeding the response to large changes in a reading. When the difference between two consecutive readings is larger than the threshold, then the reading is displayed unfiltered. When the difference between two consecutive readings drops below the threshold, readings will be filtered again.

9. Use the keypad to enter a value. The default value is 0.01.

10. Press the **Enter** key to enter the threshold.

11. Press the **Escape** key repeatedly to return to the Main menu screen.

10.4 Shutoff Time

The YSI 556 MPS shuts off automatically after 30 minutes of inactivity. The shut off time may be changed as described below.

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

2. Use the arrow keys to highlight the **Shutoff time** selection on the system setup screen. See Figure 10.2 System Setup Screen.

3. Use the keypad to enter a value from 0 to 60 minutes. The default value is 30.

   NOTE: To disable the automatic shutoff feature, enter a zero (0).

4. Press the **Enter** key to enter the correct shutoff time.

5. Press the **Escape** key repeatedly to return to the main menu screen.

10.5 Comma Radix

The user can toggle between a period (default) and comma for the radix mark by selecting this item and pressing the **Enter** key as follows:

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.
2. Use the arrow keys to highlight the **Comma radix** selection on the system setup screen. See Figure 10.2 System Setup Screen.
3. Press the **Enter** key. A check mark appears in the check box next to the comma radix selection indicating that the radix mark is a comma.

10.6 ID

This selection allows you to enter an identification name/number for your YSI 556 MPS. This ID name/number is logged in the header of each file.

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.
2. Use the arrow keys to highlight the **ID** selection. See Figure 10.1 Main Menu.
3. Use the keypad to enter an alphanumeric ID up to 15 characters in length. Refer to Section *2.9 Keypad Use*.
4. Press the **Enter** key to enter the ID.
5. Press the **Escape** key repeatedly to return to the main menu screen.

10.7 GLP Filename

This selection allows you to enter a different filename for the YSI 556 MPS Calibration Record file.

NOTE: The default filename is the "556 PC board Serial Number.glp."

6. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

7. Use the arrow keys to highlight the **GLP Filename** selection. See Figure 10.1 Main Menu.

8. Use the keypad to enter a filename up to 8 characters in length. Refer to Section *2.9 Keypad Use*.

9. Press the **Enter** key to enter the new filename.

Press the **Escape** key repeatedly to return to the main menu screen.

10.8 TDS Constant

This selection allows you to set the constant used to calculate Total Dissolved Solids (TDS). TDS in g/L is calculated by multiplying this constant times the specific conductance in mS/cm.

10.8.1 Changing the TDS Constant

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

2. Use the arrow keys to highlight the **TDS Constant** selection. See Figure 10.1 Main Menu.

3. Use the keypad to enter a value. Refer to Section *2.9 Keypad Use*. The default value is 0.65.

4. Press the **Enter** key to enter the correct TDS constant.

5. Press the **Escape** key repeatedly to return to the main menu screen.

10.9 Barometer Units

The following information is only for instruments with the barometer option.

1. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

2. Use the arrow keys to highlight the **Barometer units** selection on the system setup screen. See Figure 10.2 System Setup Screen.

3. Press the **Enter** key. The Barometer units screen will appear.

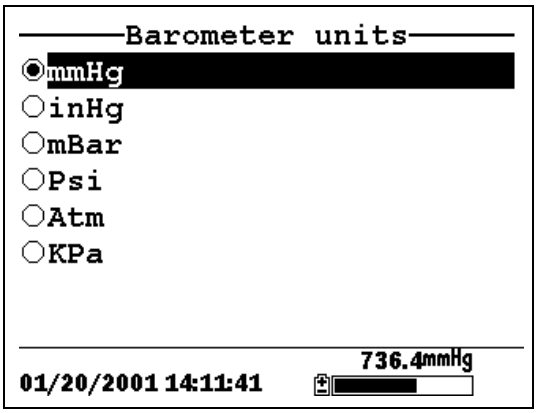


Figure 10.5 Barometer Units Screen

A black dot indicates the currently selected units.

4. Use the arrow keys to highlight your desired barometric unit.

5. Press the **Enter** key to select your choice. A black dot will appear in the circle next to your selected units.

6. Press the **Escape** key repeatedly to return to the main menu screen.

10.10 Calibrate Barometer

The optional barometer has been factory calibrated to provide accurate readings. However, some sensor drift may occur over time, requiring occasional calibration by the user, as follows:

1. Determine your local barometric pressure from an independent laboratory barometer or from your local weather service.

2. If the barometric pressure (BP) reading is from your local weather station, reverse the equation that corrects it to sea level.

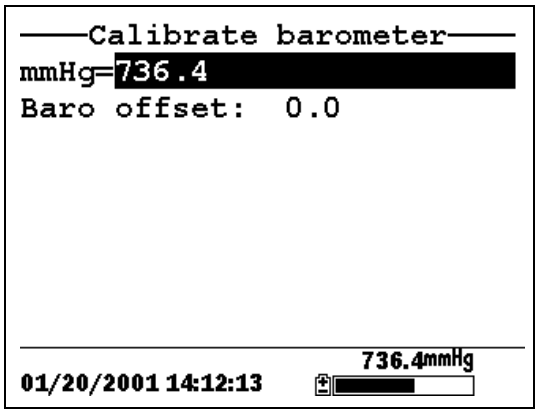
   NOTE: For this equation to be accurate, the barometric pressure units must be in mm Hg.

   True BP = (Corrected BP) – [2.5 * (Local Altitude/100)]

3. Go to the system setup screen as described in Section *10.1 Accessing the System Setup Screen*.

4. Use the arrow keys to highlight the **Calibrate barometer** selection on the system setup screen. See Figure 10.2 System Setup Screen.

5. Press the **Enter** key. The Calibrate Barometer screen is displayed.



6. Use the keypad to input the known barometric pressure value as determined in Step 2.

7. Press the **Enter** key. The new barometer reading is displayed as well as the approximate offset from the factory reading.

   NOTE: To return the sensor to the factory setting, subtract the offset amount from the current setting and repeat Steps 5 to 7.

8. Press the **Escape** key repeatedly to return to the main menu screen.

11.Maintenance

11.1 Sensor Care and Maintenance

Once the sensors have been properly installed, remember that periodic cleaning and DO membrane changes are required.

11.1.1 DO Sensor

For best results, we recommend that the KCl solution and the membrane cap be changed at least once every 30 days.

1. It is important to recognize that oxygen dissolved in the sample is consumed during sensor operation. It is therefore essential that the sample be continuously stirred at the sensor tip. If stagnation occurs, your readings will be artificially low. Stirring may be accomplished by mechanically moving the sample around the sensor tip, or by rapidly moving the sensor through the sample. The rate of stirring should be at least 1 foot per second.

2. Membrane life depends on usage. Membranes will last a long time if installed properly and treated with care. Erratic readings are a result of loose, wrinkled, damaged, or fouled membranes, or from large (more than 1/8" diameter) bubbles in the electrolyte reservoir. If erratic readings or evidence of membrane damage occurs, you should replace the membrane and the electrolyte solution. The average replacement interval is two to four weeks.

3. If the membrane is coated with oxygen consuming (e.g. bacteria) or oxygen producing organisms (e.g. algae), erroneous readings may occur.

4. Chlorine, sulfur dioxide, nitric oxide, and nitrous oxide can affect readings by behaving like oxygen at the sensor. If you suspect erroneous readings, it may be necessary to determine if these gases are the cause.

5. Avoid any environment that contains substances that may attack the probe module and sensor materials. Some of these substances are concentrated acids, caustics, and strong solvents. The sensor materials that come in contact with the

sample include FEP Teflon, acrylic plastic, EPR rubber, stainless steel, epoxy, polyetherimide and the PVC cable covering.

6. It is possible for the silver anode, which is the entire silver body of the sensor, to become contaminated. This will prevent successful calibration. To restore the anode, refer to Section *11.1.1 DO Sensor, Silver Anode Cleaning.*

7. For correct sensor operation, the gold cathode must always be bright. If it is tarnished (which can result from contact with certain gases), or plated with silver (which can result from extended use with a loose or wrinkled membrane), the gold surface must be restored. To restore the cathode, refer to Section *11.1.1 DO Sensor, Gold Cathode Cleaning.*

8. To keep the electrolyte from drying out, store the sensor in the transport/calibration cup with at least 1/8″ of water.

Silver Anode Cleaning

After extended use, a thick layer of AgCl builds up on the silver anode reducing the sensitivity of the sensor. The anode must be cleaned to remove this layer and restore proper performance. The cleaning can be chemical or mechanical:

Chemical Cleaning: Remove the membrane cap and soak the entire anode section in a 14% ammonium hydroxide solution for 2 to 3 minutes, followed by a thorough rinsing with distilled or deionized water. The anode should then be thoroughly wiped with a wet paper towel to remove the residual layer from the anode.

Mechanical Cleaning: Sand off the dark layer from the silver anode with 400 grit wet/dry sandpaper. Wrap the sandpaper around the anode and twist the sensor. Rinse the anode with clean water after sanding, followed by wiping thoroughly with a wet paper towel.

NOTE: After cleaning, a new membrane cap must be installed. Refer to Section *3.4.3 Membrane Cap Installation***.**

Turn the instrument on and allow the system to stabilize for at least 30 minutes. If, after several hours, you are still unable to calibrate, contact your dealer or YSI Customer Service. Refer to *Appendix E Customer Service*.

Gold Cathode Cleaning

For correct sensor operation, the gold cathode must be textured properly. It can become tarnished or plated with silver after extended use. The gold cathode can be cleaned by using the adhesive backed sanding disc and tool provided in the YSI 5238 Probe Reconditioning Kit.

Using the sanding paper provided in the YSI 5238 Probe Reconditioning Kit, wet sand the gold with a twisting motion about 3 times or until all silver deposits are removed and the gold appears to have a matte finish. Rinse the cathode with clean water after sanding, followed by wiping thoroughly with a wet paper towel. If the cathode remains tarnished, contact your dealer or YSI Customer Service. Refer to *Appendix E Customer Service*.

NOTE: After cleaning, a new membrane cap must be installed. Refer to Section *3.4.3 Membrane Cap Installation***.**

11.1.2 DO Sensor Replacement

1. Remove the probe sensor guard.

⚠ **CAUTION:** Thoroughly dry the sensor so that no water enters the probe module sensor port when the sensor is removed.

2. Insert the long end of the hex key wrench into the small hole in the side of the probe module bulkhead. Turn the wrench counterclockwise and remove the screw. (You do not have to remove the screw all the way to release the sensor.)

3. Pull the old DO sensor module straight out of the probe module body.

   NOTE: The DO sensor is not threaded, it is keyed, so it cannot be removed by twisting.

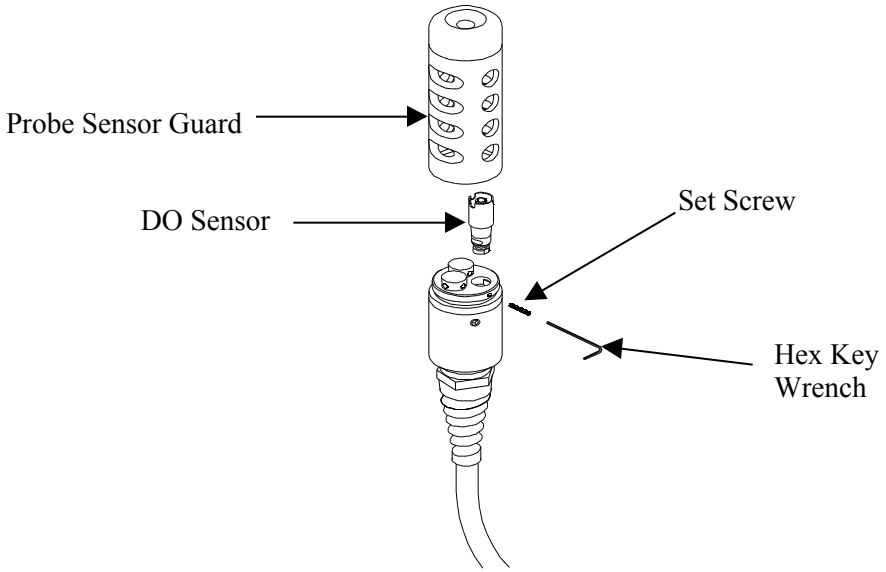


Figure 11.1 DO Sensor Replacement

4. Insert the new DO sensor module. Make sure that the inside of the probe module sensor port and the o-ring on the sensor are clean, with no contaminants, such as grease, dirt, or hair. The DO sensor is keyed, or has a flat side, so that it cannot be aligned improperly.

NOTE: Make sure the DO sensor bottoms out before the set screw is inserted.

5. Insert the set screw into the small hole in the side of the probe module bulkhead, and turn clockwise to rethread.

⚠ **CAUTION:** Make sure that you do not cross-thread the set screw. Use the hex key wrench to tighten the screw in properly, making sure that the screw does not stick out of the side of the probe module bulkhead. The probe sensor guard will not thread

on properly and damage may result if the screw is allowed to stick out.

NOTE: The YSI 5563 DO sensor is shipped dry. A shipping membrane was installed to protect the electrode. A new membrane cap must be installed before the first use. Refer to Section *3.4.1 Sensor Installation.*

11.1.3 YSI 5564 pH and 5565 Combination pH/ORP Sensor Cleaning

Cleaning is required whenever deposits or contaminants appear on the glass and/or platinum surfaces of these sensors or when the response of the sensor becomes slow.

1. Remove the sensor from the probe module.

2. Initially, simply use clean water and a soft clean cloth, lens cleaning tissue, or cotton swab to remove all foreign material from the glass bulb (YSI 5564 and YSI 5565) and platinum button (YSI 5565). Then use a moistened cotton swab to carefully remove any material that may be blocking the reference electrode junction of the sensor.

⚠ **CAUTION:** When using a cotton swab with the YSI 5564 or YSI 5565, be careful NOT to wedge the swab tip between the guard and the glass sensor. If necessary, remove cotton from the swab tip, so that the cotton can reach all parts of the sensor tip without stress.

NOTE: If good pH and/or ORP response is not restored by the above procedure, perform the following additional procedure:

1. Soak the sensor for 10-15 minutes in clean water containing a few drops of commercial dishwashing liquid.

2. GENTLY clean the glass bulb and platinum button by rubbing with a cotton swab soaked in the cleaning solution.

3. Rinse the sensor in clean water, wipe with a cotton swab saturated with clean water, and then re-rinse with clean water.

NOTE: If good pH and/or ORP response is still not restored by the above procedure, perform the following additional procedure:

1. Soak the sensor for 30-60 minutes in one molar (1 M) hydrochloric acid (HCl). This reagent can be purchased from most distributors. Be sure to follow the safety instructions included with the acid.

2. GENTLY clean the glass bulb and platinum button by rubbing with a cotton swab soaked in the acid.

3. Rinse the sensor in clean water, wipe with a cotton swab saturated with clean water, and then re-rinse with clean water. To be certain that all traces of the acid are removed from the sensor crevices, soak the sensor in clean water for about an hour with occasional stirring.

NOTE: If biological contamination of the reference junction is suspected or if good response is not restored by the above procedures, perform the following additional cleaning step:

1. Soak the sensor for approximately 1 hour in a 1 to 1 dilution of commercially available chlorine bleach.

2. Rinse the sensor with clean water and then soak for at least 1 hour in clean water with occasional stirring to remove residual bleach from the junction. (If possible, soak the sensor for period of time longer than 1 hour in order to be certain that all traces of chlorine bleach are removed.) Then re-rinse the sensor with clean water and retest.`

11.1.4 Temperature/Conductivity Sensor Cleaning

The single most important requirement for accurate and reproducible results in conductivity measurement is a clean cell. A dirty cell will change the conductivity of a solution by contaminating it. The small cleaning brush included in the YSI 5511 Maintenance Kit is ideal for this purpose.

To clean the conductivity cell:

1. Dip the brush in clean water and insert it into each hole 15-20 times.

2. Rinse the cell thoroughly in deionized or clean tap water.

NOTE: In the event that deposits have formed on the electrodes, perform the following additional procedure:

1. Use a mild detergent solution in combination with the brush. Dip the brush in the solution and insert it into each hole 15-20 times.

2. Rinse the cell thoroughly in deionized or clean tap water.

   NOTE: After cleaning, check the response and accuracy of the conductivity cell with a calibration standard.

   NOTE: If this procedure is unsuccessful, or if sensor performance is impaired, it may be necessary to return the sensor to a YSI authorized service center for service, Refer to *Appendix E Customer Service*.

   The temperature portion of the sensor requires no maintenance.

11.2 Upgrading YSI 556 MPS Software

1. Access the YSI Environmental Software Downloads page as described in *Appendix G EcoWatch* Step 1 through 3.

2. Click on the **YSI Instruments Software Updates** link (or scroll down until you see YSI 556 MPS).

3. Click on the file icon to the right of the **YSI 556 MPS** listing and save the file to a temporary directory on your computer.

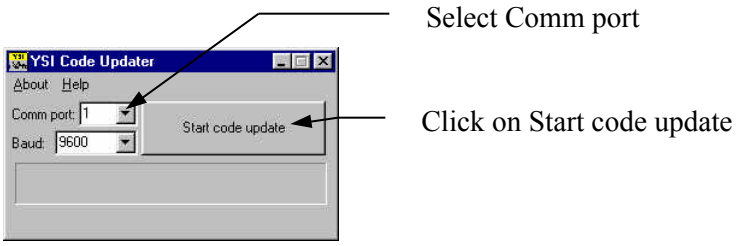
4. After the download is complete, run the file (that you just downloaded) and follow the on screen instructions to install the YSI Code Updater on your computer. If you encounter difficulties, contact YSI customer service for advice. Refer to *Appendix E Customer Service*.

5. If necessary, disconnect the YSI 5563 Probe Module from the YSI 556 MPS instrument.

6. Connect the YSI 556 MPS to a serial port of your computer via the 655173 PC interface cable. See Figure 8.6 Computer/Instrument Interface.

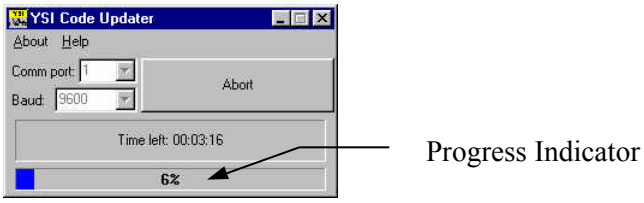
7. Press the **On/off** key on the YSI 556 MPS to display the run screen.

8. Run the YSI Code Updater software that you just installed on your computer. The following window will be displayed:

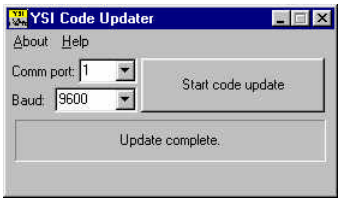


9. Set the Comm port number to match the port that you connected the 655173 PC Interface Cable to, then click on the **Start Code Update button**.

The YSI 556 MPS screen will blank out and a progress indicator will be displayed on the PC.



When the update is finished (indicated on the PC screen), the YSI 556 MPS will return to the Run screen. See Figure 7.1 Run Screen.



10. Close the YSI Code Updater window (on the PC) by clicking on the "X" in the upper right corner of the window.

11. Disconnect the YSI 556 MPS from the 655173 PC interface cable and reconnect it to the YSI 5563 Probe Module. Refer to Section *3.6 Instrument/Cable Connection*.

12.Storage

Proper storage between periods of usage will not only extend the life of the sensors, but will also ensure that the unit will be ready to use as quickly as possible in your next application.

12.1 General Recommendations for Short Term Storage

No matter what sensors are installed in the instrument, it is important to keep them moist without actually immersing them in liquid. Immersing them could cause some of them to drift or result in a shorter lifetime.

YSI recommends that short term storage of all multi-parameter instruments be done by placing approximately 1/2 inch of tap water in the transport/calibration cup that was supplied with the instrument, and by placing the probe module with all of the sensors installed into the cup. The use of a moist sponge instead of a 1/2 inch of tap water is also acceptable, as long as its presence does not compromise the attachment of the cup to the probe module. The transport/calibration cup should be sealed to prevent evaporation.

NOTE: Ensure that an o-ring is installed in the o-ring groove on the threaded end of the probe module body. See Figure 3.7 Transport/Calibration Cup Installation.

⚠ **CAUTION:** The water level has to be low enough so that none of the sensors are actually under water. Check the transport/calibration cup periodically to make certain that the water is still present or the sponge is still moist.

NOTE: If the storage water (tap water) is accidentally lost during field use, environmental water can be used.

12.2 General Recommendations for Long Term Storage

12.2.1 Probe Module Storage

1. Remove the pH or pH/ORP sensor from the probe module and store according to the individual sensor storage instructions found in Section *12.2.2 Sensor Storage*.

2. Seal the empty port with the provided port plug.

NOTE: Leave the conductivity/temperature sensor and dissolved oxygen sensor, with membrane cap still on, in the probe module.

3. Place 1/2″ of water, deionized, distilled or tap, in the transport/calibration cup.

⚠ **CAUTION:** The water level has to be low enough so that none of the sensors are actually under water. Check the transport/calibration cup periodically to make certain that the water is still present or the sponge is still moist.

4. Insert the probe module into the cup.

NOTE: Ensure that an o-ring is installed in the o-ring groove on the threaded end of the probe module body. See Figure 3.7 Transport/Calibration Cup Installation.

12.2.2 Sensor Storage

Temperature/Conductivity Sensor

No special precautions are required. Sensor can be stored dry or wet, as long as solutions in contact with the thermistor and conductivity electrodes are not corrosive (for example, chlorine bleach). However, it is recommended that the sensor be cleaned with the provided brush prior to long term storage. Refer to Section *11.1.4 Temperature/Conductivity Sensor Cleaning.*

pH and Combination pH/ORP Sensor

The key to sensor storage is to make certain that the reference electrode junction does not dry out. Junctions which have been allowed to dry out due to improper storage procedures can usually be rehydrated by soaking the sensor for several hours (overnight is recommended) in a solution which is 2 molar in potassium chloride. If potassium chloride solution is not available, soaking the sensor in tap water or commercial pH buffers may restore sensor function. However in some cases the sensor may have been irreparably damaged by the dehydration and will require replacement.

⚠ **CAUTION:** Do not store the sensor in distilled or deionized water as the glass sensor may be damaged by exposure to this medium.

1. Remove the pH or pH/ORP sensor from the probe module.

2. Seal the empty port with the provided port plug.

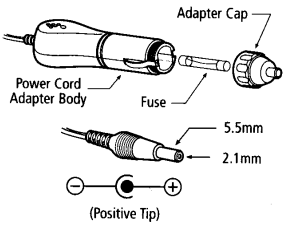
3. Place the sensor in the storage vessel (plastic boot or bottle) which was on the sensor at delivery. The vessel should contain a solution which is 2 molar in potassium chloride.

   NOTE: Make certain that the vessel is sealed to prevent evaporation of the storage solution.

13.Troubleshooting

The following sections describe problems you may encounter when using the YSI 556 MPS and provides suggestions to overcome the symptom.

PROBLEM	POSSIBLE SOLUTION
Display Problems	
No display is visible after pressing the on/off key.	If C cells are used, make certain that they are installed properly with regard to polarity and that good batteries are used. If a rechargeable battery pack is used, place the pack in the instrument and charge for 30 minutes.
Instrument software appears to be locked up as evidenced by no response to keypad entries or display not changing.	First, attempt to reset the instrument by simply turning off and then on again. If this fails, remove battery power from the instrument for 30 seconds and then reapply power. When using C cells, remove the battery lid and one of the batteries; when using the rechargeable battery pack, remove the pack completely from the instrument. After 30 seconds replace the battery or battery pack and check for instrument function.
The 556 display flashes and the instrument speaker makes a continuous clicking sound.	The battery voltage is low. Change to new C cells or recharge the 6117 battery pack.
Water Damage to Instrument	
Leakage detected in battery compartment when using C cells	Dispose of batteries properly. Dry the battery compartment using compressed air if possible. If corrosion is present on battery terminals, contact YSI Customer Service.
Water has contacted rechargeable battery pack	Remove battery pack immediately. Send battery pack to YSI Product Service for evaluation. **CAUTION**: DO NOT REUSE BATTERY PACK UNTIL YSI PRODUCT SERVICE HAS EVALUATED IT.
Leakage suspected into the main cavity of the instrument case	Remove the batteries immediately. Return the instrument to YSI Product Service.

PROBLEM	POSSIBLE SOLUTION
Optional Cigarette Lighter Charger	
Power cord fuse blown 	1. Unscrew adapter's cap, remove tip and pull out fuse. 2. Replace fuse with a new 2-amp fast-blow fuse from an electronics store such as Radio Shack. 3. Reassemble the adapter and securely screw the cap back onto the adapter body.
File Problems	
Upload of files from YSI 556 MPS to PC fails	1. Make sure that cable is connected properly to both 556 and PC. 2. Make certain that the proper Comm port is selected in EcoWatch for Windows.
Barometer data is not stored with sensor data file.	Make sure **Store barometer** is active in the 556 **Logging setup** menu.
Site Descriptions in the **Site List** are "grayed-out" and not available for appending files with additional data.	There is a parameter mismatch between the current 556 setup and that initially used. Change the current logging and sensor setup to match the setup that was initially used to create the file.
Sensor Problems	
Dissolved Oxygen reading unstable or inaccurate. Out of Range message appears during calibration.	Sensor not properly calibrated. Follow DO cal procedures.
	Membrane not properly installed or may be punctured. Replace membrane cap.
	DO sensor electrodes require cleaning. Follow DO cleaning procedure. Use 5511 Maintenance kit.
	Water in sensor connector. Dry connector; reinstall sensor.
	Algae or other contaminant clinging to DO sensor. Rinse DO sensor with clean water.
	Barometric pressure entry is incorrect. Repeat DO cal procedure.
	Calibrated at extreme temperature. Recalibrate at (or near) sample temperature.
	DO sensor has been damaged. Replace sensor.
	Internal failure. Return probe module for service.

PROBLEM	POSSIBLE SOLUTION
Sensor Problems	
pH or ORP readings are unstable or inaccurate. Out of Range message appears during calibration.	Sensor requires cleaning. Follow sensor cleaning procedure.
	Sensor requires calibration. Follow cal procedures.
	pH sensor reference junction has dried out from improper storage. Soak sensor in tap water or buffer until readings become stable.
	Water in sensor connector. Dry connector; reinstall sensor.
	Sensor has been damaged. Replace sensor.
	Calibration solutions out of spec or contaminated with other solution. Use new calibration solutions.
	ORP fails Zobell check. Take into account temperature dependence of Zobell solution readings.
	Internal failure. Return probe module for service.
Conductivity unstable or inaccurate. Out of Range message appears during calibration.	Conductivity improperly calibrated. Follow calibration procedure.
	Conductivity sensor requires cleaning. Follow cleaning procedure.
	Conductivity sensor damaged. Replace sensor.
	Calibration solution out of spec or contaminated. Use new calibration solution.
	Internal failure. Return probe module for service.
	Calibration solution or sample does not cover entire sensor. Immerse sensor fully.
Temperature, unstable or inaccurate	Water in connector. Dry connector; reinstall sensor.
	Sensor has been damaged. Replace the 5560 sensor.
Installed sensor has no reading	The sensor has been disabled. Enable sensor.
	Water in sensor connector. Dry connector; reinstall sensor.
	Sensor has been damaged. Replace the sensor.
	Report output improperly set up. Set up report output.
	Internal failure. Return probe module for service.

If these guidelines and tips fail to correct your problem or if any other symptoms occur, contact YSI Customer Service for Advice. Refer to *Appendix E Customer Service*.

14. Appendix A YSI 556 MPS Specifications

14.1 Sensor Specifications

Dissolved Oxygen		
Sensor Type		Steady state polarographic
Range:	*% air sat'n* *mg/L*	_ 0 to 500% air saturation _ 0 to 50 mg/L
Accuracy:	*% air sat'n* *mg/L*	_ 0 to 200% air saturation: ±2% of the reading or 2% air saturation; whichever is greater _ 200 to 500% air saturation: ±6% of the reading _ 0 to 20 mg/L: ±2% of the reading or 0.2 mg/L; whichever is greater _ 20 to 50 mg/L: ±6% of the reading
Resolution:	*% air sat'n* *mg/L*	_ 0.1% air saturation _ 0.01 mg/L
Temperature		
Sensor Type:		YSI Precision™ thermistor
Range:		-5 to 45°C
Accuracy:		±0.15°C
Resolution:		0.01°C
Conductivity		
Sensor Type:		4-electrode cell with auto-ranging
Range:		0 to 200 mS/cm
Accuracy:		±0.5% of reading or ±0.001 mS/cm; whichever is greater–4 meter cable ±1.0% of reading or ±0.001 mS/cm; whichever is greater–20 meter cable
Resolution:		0.001 mS/cm to 0.1 mS/cm (range-dependent)
Salinity		
Sensor Type:		Calculated from conductivity and temperature
Range:		0 to 70 ppt
Accuracy:		±1.0% of reading or 0.1 ppt; whichever is greater
Resolution:		0.01 ppt

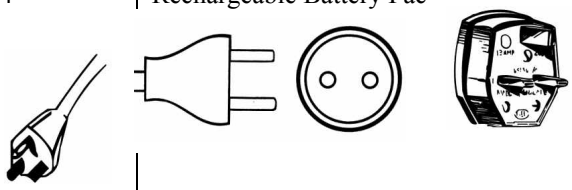
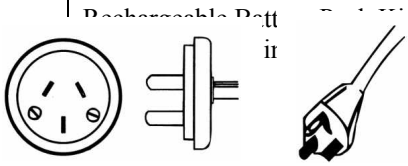
pH (optional)	
Sensor Type:	Glass combination electrode
Range:	0 to 14 units
Accuracy:	±0.2 units
Resolution:	0.01 units
ORP (optional)	
ORP Sensor Type:	Platinum button
Range:	-999 to +999 mV
Accuracy:	±20 mV
Resolution:	0.1 mV

Barometer (optional)	
Range:	500 to 800 mm Hg
Accuracy:	±3 mm Hg within ±15°C temperature range from calibration point
Resolution:	0.1 mm Hg

14.2 Instrument Specifications

Memory Size:	1.5 MB Flash Memory 49,000 data sets (@ 6 parameters per set plus time stamp) 100 Sites
Size:	11.9 cm width x 22.9 cm length (4.7 in. x 9 in.)
Weight with batteries:	0.92 kg (2.1 lbs)
Power:	4 alkaline C-cells; optional rechargeable pack
Cables:	4, 10, and 20 m (13.1, 32.8, 65.6 ft.) lengths
Warranty:	3-Years for the instrument; 1-Year for the probe modules and cable

15.Appendix B Instrument Accessories

ITEM #	ACCESSORY
5563-4	4m Cable with DO/temp/conductivity
5563-10	10m Cable with DO/temp/conductivity
5563-20	20m Cable with DO/temp/conductivity
5564	pH Kit
5565	pH/ORP Kit
6118	Rechargeable Battery Pack Kit for use in US
5094	Rechargeable Battery Pack Kit with universal charger and three adapter s 
5095	Rechargeable Batt Pack Kit with universal charger and two adapter in plications 
5083	Flow Cell – probe module is secured in the flow cell and groundwater is pumped through it
616	Charger, Cigarette Lighter – used to power up the instrument from a car's cigarette lighter
4654	Tripod
614	Ultra Clamp, C Clamp –used to clamp the instrument to a table top or car dashboard
6081	Large Carrying Case, Hard-sided
5085	Hands-free Harness
5065	Carrying Case, Form-fitted, for use in the field – has a clear vinyl window, shoulder strap, belt loop strap and hand strap

16.Appendix C Required Federal Communications Notice

The Federal Communications Commission defines this product as a computing device and requires the following notice.

This equipment generates and uses radio frequency energy and if not installed and used properly, may cause interference to radio and television reception. It has been type tested and found to comply with the limits for a Class A or Class B computing device in accordance with the specification in Subpart J of Part 15 of FCC Rules, which are designed to provide reasonable protection against such interference in a residential installation. However, there is no guarantee that interference will not occur in a particular installation. If this equipment does cause interference to radio or television reception, which can be determined by turning the equipment off and on, the user is encouraged to try to correct the interference by one or more of the following measures:

- Reorient the receiving antenna
- Relocate the computer with respect to the receiver
- Move the computer away from the receiver
- Plug the computer into a different outlet so that the computer and receiver are on different branch circuits.

If necessary, the user should consult the dealer or an experienced radio/television technician for additional suggestions. The user may find the following booklet, prepared by the Federal Communications Commission, helpful: "How to Identify and Resolve Radio-TV Interference Problems". This booklet is available from the U.S. Government Printing Office, Washington, D.C. 20402, Stock No.0004-000-00345-4.

YSI Conductivity solutions: 3161, 3163, 3165, 3167, 3168, 3169

INGREDIENTS:
- Iodine
- Potassium Chloride
- Water

WARNING: INHALATION MAY BE FATAL.

⚠ ***CAUTION*: AVOID INHALATION, SKIN CONTACT, EYE CONTACT OR INGESTION. MAY EVOLVE TOXIC FUMES IN FIRE.**

Harmful if ingested or inhaled. Skin or eye contact may cause irritation. Has a corrosive effect on the gastro-intestinal tract, causing abdominal pain, vomiting, and diarrhea. Hyper-sensitivity may cause conjunctivitis, bronchitis, skin rashes etc. Evidence of reproductive effects.

FIRST AID:
INHALATION: Remove victim from exposure area. Keep victim warm and at rest. In severe cases seek medical attention.
SKIN CONTACT: Remove contaminated clothing immediately. Wash affected area thoroughly with large amounts of water. In severe cases seek medical attention.
EYE CONTACT: Wash eyes immediately with large amounts of water, (approx. 10 minutes). Seek medical attention immediately.
INGESTION: Wash out mouth thoroughly with large amounts of water and give plenty of water to drink. Seek medical attention immediately.

YSI pH 4.00, 7.00, and 10.00 Buffer Solutions: 3821, 3822, 3823

pH 4 INGREDIENTS:

- Potassium Hydrogen Phthalate
- Formaldehyde
- Water

pH 7 INGREDIENTS:

- Sodium Phosphate, Dibasic
- Potassium Phosphate, Monobasic
- Water

pH 10 INGREDIENTS:

- Potassium Borate, Tetra
- Potassium Carbonate
- Potassium Hydroxide
- Sodium (di) Ethylenediamine Tetraacetate
- Water

⚠ ***CAUTION*** **- AVOID INHALATION, SKIN CONTACT, EYE CONTACT OR INGESTION. MAY AFFECT MUCOUS MEMBRANES.**

Inhalation may cause severe irritation and be harmful. Skin contact may cause irritation; prolonged or repeated exposure may cause Dermatitis. Eye contact may cause irritation or conjunctivitis. Ingestion may cause nausea, vomiting and diarrhea.

FIRST AID:

INHALATION - Remove victim from exposure area to fresh air immediately. If breathing has stopped, give artificial respiration. Keep victim warm and at rest. Seek medical attention immediately.

SKIN CONTACT - Remove contaminated clothing immediately. Wash affected area with soap or mild detergent and large amounts of water (approx. 15-20 minutes). Seek medical attention immediately.

EYE CONTACT - Wash eyes immediately with large amounts of water (approx. 15-20 minutes), occasionally lifting upper and lower lids. Seek medical attention immediately.

INGESTION - If victim is conscious, immediately give 2 to 4 glasses of water and induce vomiting by touching finger to back of throat. Seek medical attention immediately.

YSI Zobell Solution: 3682

INGREDIENTS:
- Potassium Chloride
- Potassium Ferrocyanide Trihydrate
- Potassium Ferricyanide

⚠ ***CAUTION*** **- AVOID INHALATION, SKIN CONTACT, EYE CONTACT OR INGESTION. MAY AFFECT MUCOUS MEMBRANES.**

May be harmful by inhalation, ingestion, or skin absorption. Causes eye and skin irritation. Material is irritating to mucous membranes and upper respiratory tract. The chemical, physical, and toxicological properties have not been thoroughly investigated.

Ingestion of large quantities can cause weakness, gastrointestinal irritation and circulatory disturbances.

FIRST AID:

INHALATION - Remove victim from exposure area to fresh air immediately. If breathing has stopped, give artificial respiration. Keep victim warm and at rest. Seek medical attention immediately.

SKIN CONTACT - Remove contaminated clothing immediately. Wash affected area with soap or mild detergent and large amounts of water (approx. 15-20 minutes). Seek medical attention immediately.

EYE CONTACT - Wash eyes immediately with large amounts of water (approx. 15-20 minutes), occasionally lifting upper and lower lids. Seek medical attention immediately.

INGESTION - If victim is conscious, immediately give 2 to 4 glasses of water and induce vomiting by touching finger to back of throat. Seek medical attention immediately.

18.Appendix E Customer Service

For information on Customer Service Centers, refer to *Authorized Service Centers* in this appendix.

Equipment exposed to biological, radioactive, or toxic materials must be cleaned and disinfected before being returned or presented for service. A cleaning certificate must accompany the equipment. Refer to *18.2 Cleaning Instructions* in this appendix.

18.1 YSI Environmental Authorized Service Centers

International Service Centers

YSI Incorporated · Repair Center · 1725 Brannum Lane
Yellow Springs, Ohio · 45387 · Phone: (937) 767-7241
E-Mail: support@ysi.com

Hydrodata Services (UK) Ltd. · Unit 8 · Business Centre West
Avenue One · Letchworth · Herts · SG6 2HB
Phone: (44-1462) 673581 · Fax: (44-1462) 673582
Email: hydrodatauk@cs.com

YSI Nanotech · Kaizuka 1-15-4, Kawasaki-Ku · Kawaskaki
City · Japan · 210-0014 · Phone: 011-814-4222-0009
Fax: 011-81-44-221102 · E-mail: Nanotech@ysi.com

Nortech GSI · 1131 Derry Road East · Mississauga, ONT
L5T 1P3 · Canada · Phone: 800-263-3427 · Fax: 905-564-4700

US Service Centers for Water Quality and 6-Series Instruments

Ohio
YSI Incorporated · Repair Center · 1725 Brannum Lane
Yellow Springs, Ohio · 45387 · Phone: (800) 765-4974
(937) 767-7241· E-Mail: info@ysi.com

California
EQUIPCO Sales and Service · 1110 Burnett Avenue, Suite D
Concord, CA · 94520 · Phone: (800)550-5875
Fax: (510)674-8655

Colorado
Ted D. Miller Associates, Inc. · 2525 S. Wadsworth Blvd., Suite 300 · Lakewood, CO · 80227 · Phone: (303) 989-7737 Fax: (303) 989-8875 · E:mail: tdma@earthlink.net

Mississippi
C.C. Lynch & Associates, Inc. · P.O. Box 456 · 300 Davis Street · Pass Christian, MS · 39571 Phone: (800) 333-2252 (228) 452-4612 · Fax (228) 452-2563

US Service Centers for Water Quality Instruments Only

Florida
Aquatic Eco Systems, Inc. · 1767 Benbow Court · Apopka, Florida · Phone: (407) 886-3939 · Fax: (407) 886-6787

Maine
Q.C. Services · P.O. Box 68 · Harrison, Maine · 04040 Phone: (207) 583-2980

Mississippi
Aquacenter · 166 Seven Oaks Road · Leland, Mississippi 38756 · Phone: (601) 378-2861 · Fax: (601) 378-2862

Oregon
Q.C. Services · P.O. Box 14831 · Portland, Oregon · 97293 Phone: (503) 236-2712

Wisconsin
North Central Labs · 400 Lyons Road · Birnamwood, Wisconsin · Phone: (800) 648-7836 · Fax: (715) 449-2454

18.2 Cleaning Instructions

Equipment exposed to biological, radioactive, or toxic materials must be cleaned and disinfected before being serviced. Biological contamination is presumed for any instrument, probe, or other device that has been used with body fluids or tissues, or with wastewater. Radioactive contamination is presumed for any instrument, probe or other device that has been used near any radioactive source.

If an instrument, probe, or other part is returned or presented for service without a Cleaning Certificate, and if in our opinion it represents a potential biological or radioactive hazard, our service personnel reserve the right to withhold service until appropriate cleaning, decontamination, and certification has been completed. We will contact the sender for instructions as to the disposition of the equipment. Disposition costs will be the responsibility of the sender.

When service is required, either at the user's facility or at a YSI Service Center, the following steps must be taken to ensure the safety of service personnel.

- In a manner appropriate to each device, decontaminate all exposed surfaces, including any containers. 70% isopropyl alcohol or a solution of 1/4-cup bleach to 1-gallon tap water is suitable for most disinfecting. Instruments used with wastewater may be disinfected with .5% Lysol if this is more convenient to the user.
- The user shall take normal precautions to prevent radioactive contamination and must use appropriate decontamination procedures should exposure occur.
- If exposure has occurred, the customer must certify that decontamination has been accomplished and that no radioactivity is detectable by survey equipment.
- Any product being returned to the YSI Repair Center should be packed securely to prevent damage.
- Cleaning must be completed and certified on any product before returning it to YSI.

18.3 Packing Procedure

- Clean and decontaminate items to ensure the safety of the handler.
- Complete and include the Cleaning Certificate.
- Place the product in a plastic bag to keep out dirt and packing material.
- Use a large carton, preferably the original, and surround the product completely with packing material.
- Insure for the replacement value of the product.

18.4 Cleaning Certificate

Organization _____

Department _____

Address _____

City _____ State __ Zip _____

Country _____ Phone _____

Model No. of Device __ Lot Number _____

Contaminant (if known) _____

Cleaning Agent(s) used _____

Radioactive Decontamination Certified?

(Answer only if there has been radioactive exposure)

_____ Yes _____ No

Cleaning Certified By _____

Name Date

18.5 Warranty

The instrument is warranted for three years against defects in workmanship and materials when used for its intended purposes and maintained according to instructions. The probe module and cables are warranted for one year. The dissolved oxygen, temperature/conductivity, pH, and pH/ORP combination sensors are warranted for one year. Damage due to accidents, misuse, tampering, or failure to perform prescribed maintenance is not covered. The warranty period for chemicals and reagents is determined by the expiration date printed on their labels. Within the warranty period, YSI will repair or replace, at its sole discretion, free of charge, any product that YSI determines to be covered by this warranty.

To exercise this warranty, write or call your local YSI representative, or contact YSI Customer Service in Yellow Springs, Ohio. Send the product and proof of purchase, transportation prepaid, to the Authorized Service Center selected by YSI. Repair or replacement will be made and the product returned transportation prepaid. Repaired or replaced products are warranted for the balance of the original warranty period, or at least 90 days from date of repair or replacement.

Limitation of Warranty

This Warranty does not apply to any YSI product damage or failure caused by (i) failure to install, operate or use the product in accordance with YSI's written instructions, (ii) abuse or misuse of the product, (iii) failure to maintain the product in accordance with YSI's written instructions or standard industry procedure, (iv) any improper repairs to the product, (v) use by you of defective or improper components or parts in servicing or repairing the product, or (vi) modification of the product in any way not expressly authorized by YSI.

THIS WARRANTY IS IN LIEU OF ALL OTHER WARRANTIES, EXPRESSED OR IMPLIED, INCLUDING ANY WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE. YSI'S LIABILITY UNDER THIS WARRANTY IS LIMITED TO REPAIR OR REPLACEMENT OF THE PRODUCT, AND THIS SHALL BE YOUR SOLE AND EXCLUSIVE REMEDY FOR ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY. IN NO EVENT SHALL YSI BE LIABLE FOR ANY SPECIAL, INDIRECT, INCIDENTAL OR CONSEQUENTIAL DAMAGES RESULTING FROM ANY DEFECTIVE PRODUCT COVERED BY THIS WARRANTY.

19.Appendix F Ferrite Bead Installation

⚠ **WARNING:** If you are using your YSI 556 in a European Community (CE) country or in Australia or New Zealand, you must attach a ferrite bead to the 655173 PC Interface Cable and the YSI 6117 Charger Adapter Cable in order to comply with the Residential, Commercial and Light Industrial Class B Limits for radio-frequency emissions specified in EN55011 (CISPR11) for Industrial, Scientific and Medical laboratory equipment. These ferrite assemblies are supplied as part of cable kits.

1. Make a small loop (approximately 5 cm in diameter) in the cable near the YSI 556 MS-19 connector.

2. Lay the open ferrite bead assembly under the loop with the cable cross-over position within the cylinder of the ferrite bead.

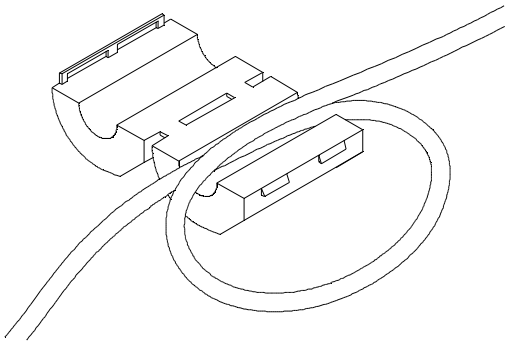


Figure 19.1 Ferrite Bead Installation

3. Snap the two pieces of the bead together making certain that the tabs lock securely.

4. When the installation is complete, the 655173 and YSI 6117 cables should resemble the following drawings.

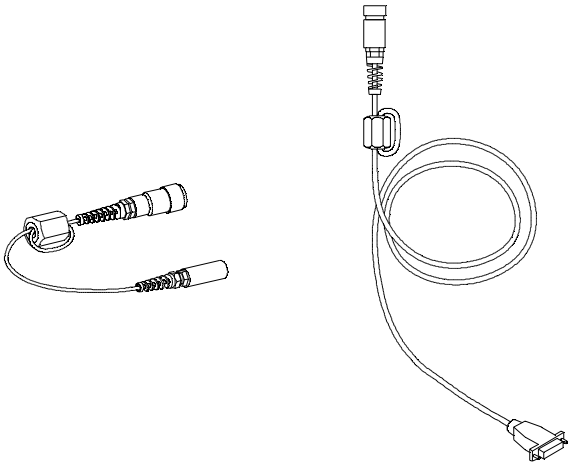


Figure 19.2 Cables with Ferrite Beads

20.Appendix G EcoWatch

EcoWatch™ for Windows™ must be used as the PC software interface to the YSI 556 MPS. EcoWatch is a powerful tool that can also be used with YSI 6-series sondes. Many features of the software will only be utilized by advanced users or are not relevant to the 556 MPS at all. This section is designed in tutorial format to familiarize you with the commonly used features of EcoWatch so that it will be possible to:

- Upload data from a 556 MPS to a PC
- Assemble plots and reports of your data
- Zoom in on certain segments of the plots of your data to facilitate analysis
- Show statistical data for your studies
- Export data in spreadsheet-compatible formats
- Print plots and reports

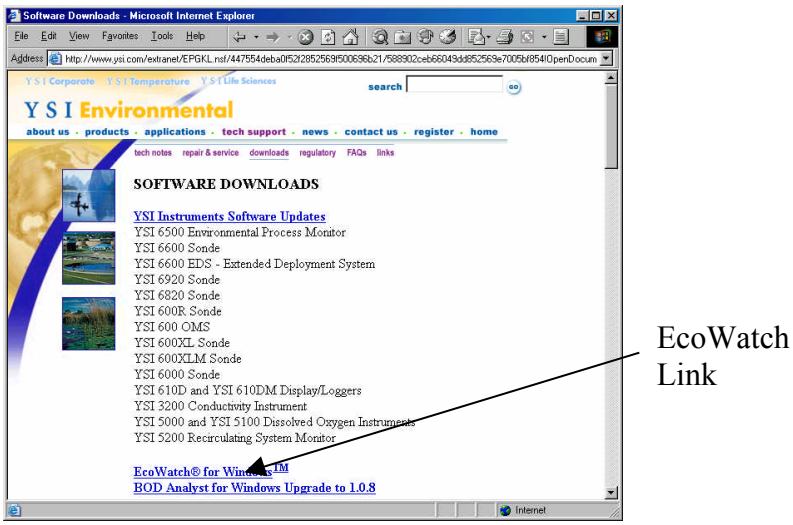
The advanced features of EcoWatch can be explored by downloading a 6-series manual from the YSI Web Site (www.ysi.com), purchasing a hard copy of the manual through YSI Customer Service (Item # 069300), or utilizing the on-line help feature of the software.

20.1 Installing EcoWatch for Windows

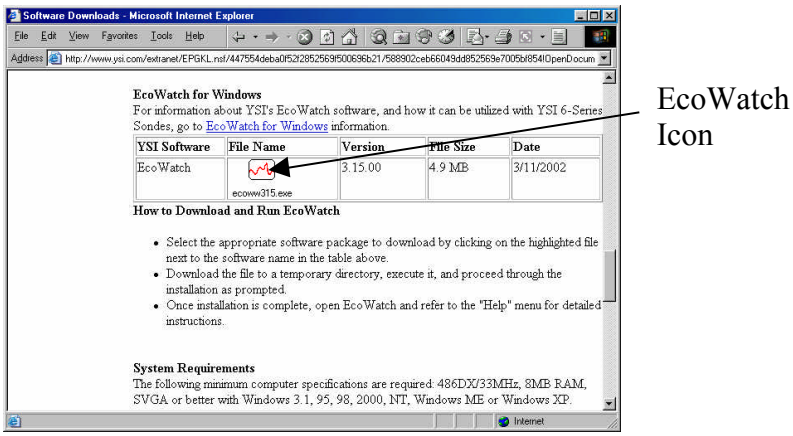
EcoWatch for Windows is available at no cost via a download from the YSI Web Site.

1. Access the YSI Environmental Web Site at www.ysi.com/edownloads.

2. Click on the **EcoWatch for Windows** link (or scroll down until you see the EcoWatch for Windows icon).



3. Click on the EcoWatch for Windows icon and save the file to a temporary directory on your computer.



4. After the download is complete, run the EcoWatch file (that you just downloaded) and follow the on screen instructions to install the software on your computer.

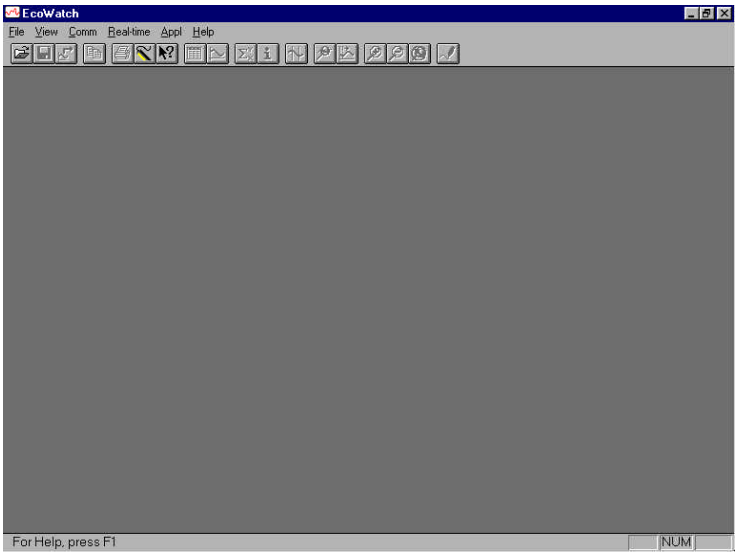
If you encounter difficulties in the download procedure, contact YSI Customer Service. Refer to ***Appendix E Customer Service***. Alternatively, you may purchase the software on CD ROM (Item #006075) by contacting YSI Customer Service.

20.2 EcoWatch Tutorial

This EcoWatch tutorial is designed to teach you the commonly used operations associated with the software when used with your 556 MPS.

After you have uploaded a file, Refer to Section 8.4 Upload to PC, you will see two files in the C:\ECOWWIN\DATA directory; the file you transferred and a file supplied by YSI designated SAMPLE.DAT. This SAMPLE.DAT file is referred to in the remainder of this tutorial section. After following the instructions below for the analysis of SAMPLE.DAT, you apply the same analysis to the data file which was uploaded from your 556 MPS to assure that you are familiar with the basic features and capabilities of EcoWatch for Windows.

To start the analysis of the SAMPLE.DAT file, note that a shortened menu bar is visible and many of the tools in the toolbar appear dimmed or "grayed out" before any file is opened (see below).



Full activation of EcoWatch features will be realized after a file is opened.

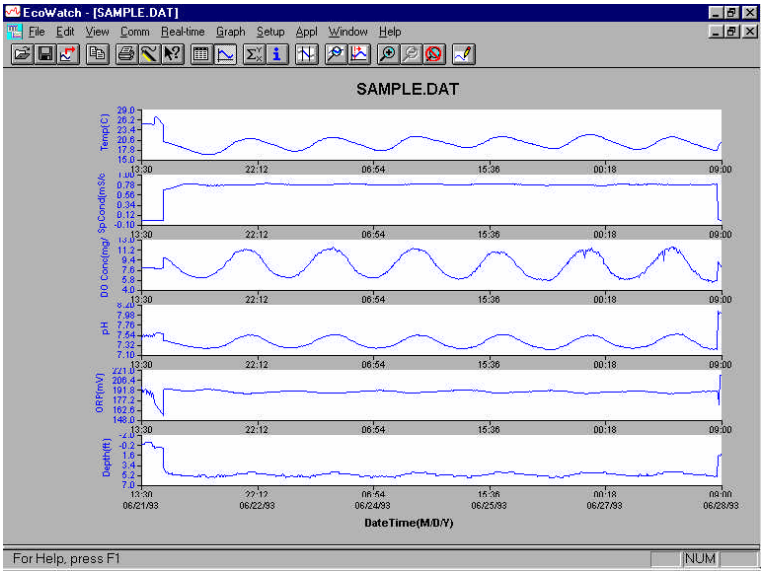
To open the sample data file:

5. 1. Click the **File** menu button in the toolbar.

6. 2. Select the **SAMPLE.DAT** file.

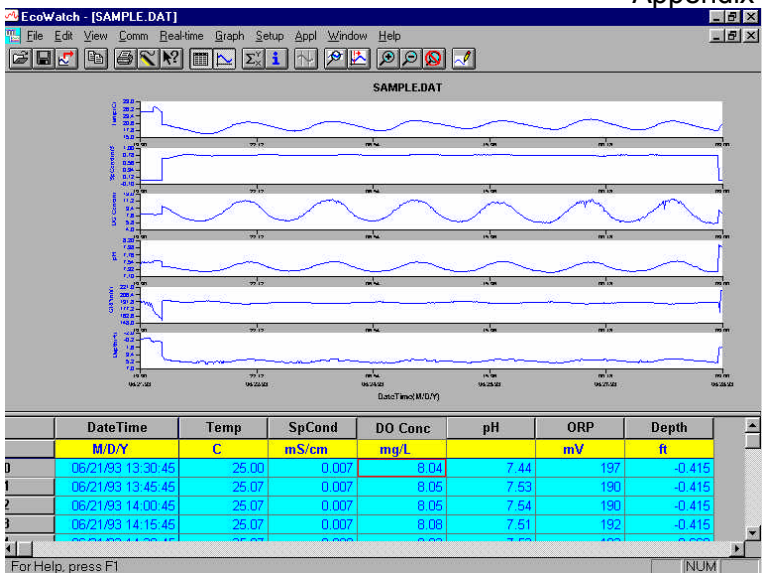
7. 3. Click **OK** to open the file.

The following display will appear:



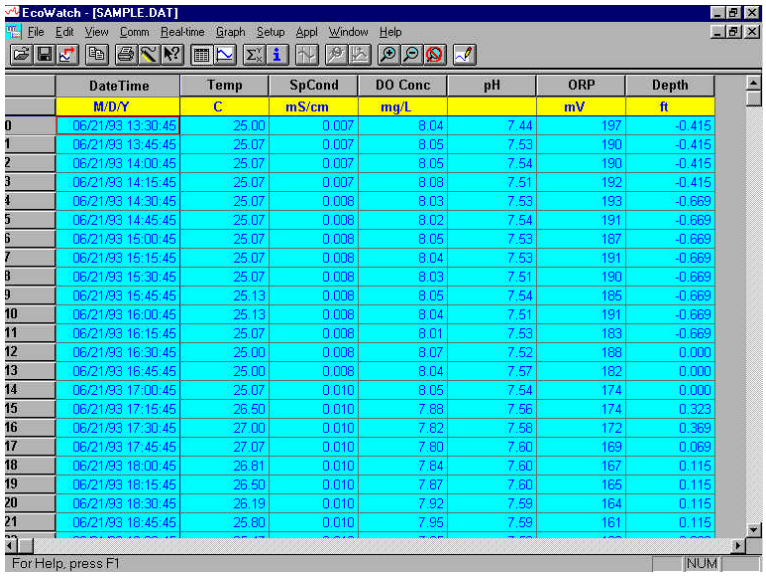
Note that the data in this file appears as a graph of temperature, specific conductance, dissolved oxygen, pH, ORP, and depth, all versus time. The graphs are scaled automatically so that all data fits comfortably on the computer screen. Note also that this data file was obtained with a 6-series sonde for which a depth sensor is available. Depth is NOT a current parameter for the 556 MPS.

The **Table** and **Graph** buttons on the toolbar are on/off switches that are used to display or hide the graph and table pages respectively. When displaying a graph and a table at the same time, you can control the relative size of the two pages by placing the cursor over the small bar that separates them and then dragging it to the desired location. Click the **Table** button to generate the following dual display of data.



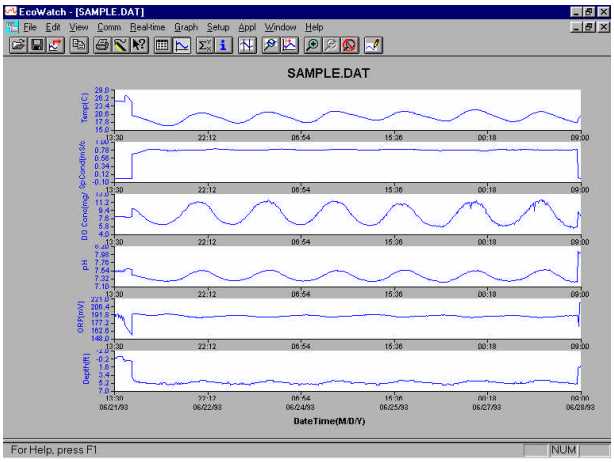
	DateTime	Temp	SpCond	DO Conc	pH	ORP	Depth
	M/D/Y	C	mS/cm	mg/L		mV	ft
0	06/21/93 13:30:45	25.00	0.007	8.04	7.44	197	-0.415
1	06/21/93 13:45:45	25.07	0.007	8.05	7.53	190	-0.415
2	06/21/93 14:00:45	25.07	0.007	8.05	7.54	190	-0.415
3	06/21/93 14:15:45	25.07	0.007	8.08	7.51	192	-0.415

Now click the **Graph** button (turn it off) to display only a report of your data as shown below. Note that the size of the report can be varied by clicking on the and buttons in the Toolbar.

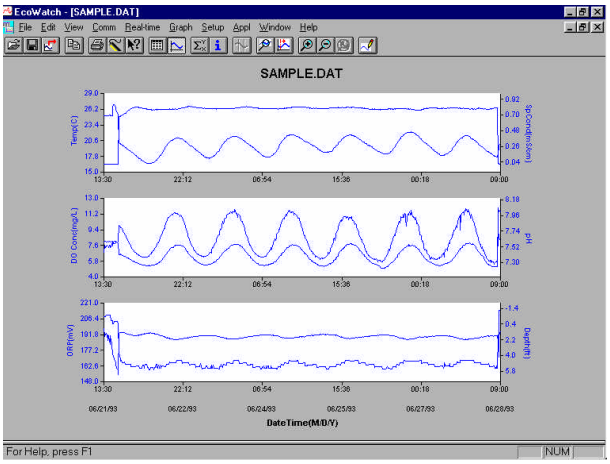


	DateTime	Temp	SpCond	DO Conc	pH	ORP	Depth
	M/D/Y	C	mS/cm	mg/L		mV	ft
0	06/21/93 13:30:45	25.00	0.007	8.04	7.44	197	-0.415
1	06/21/93 13:45:45	25.07	0.007	8.05	7.53	190	-0.415
2	06/21/93 14:00:45	25.07	0.007	8.05	7.54	190	-0.415
3	06/21/93 14:15:45	25.07	0.007	8.08	7.51	192	-0.415
4	06/21/93 14:30:45	25.07	0.008	8.03	7.53	193	-0.669
5	06/21/93 14:45:45	25.07	0.008	8.02	7.54	191	-0.669
6	06/21/93 15:00:45	25.07	0.008	8.05	7.53	187	-0.669
7	06/21/93 15:15:45	25.07	0.008	8.04	7.53	191	-0.669
8	06/21/93 15:30:45	25.07	0.008	8.03	7.51	190	-0.669
9	06/21/93 15:45:45	25.13	0.008	8.05	7.54	185	-0.669
10	06/21/93 16:00:45	25.13	0.008	8.04	7.51	191	-0.669
11	06/21/93 16:15:45	25.07	0.008	8.01	7.53	183	-0.669
12	06/21/93 16:30:45	25.00	0.008	8.07	7.52	188	0.000
13	06/21/93 16:45:45	25.00	0.008	8.04	7.57	182	0.000
14	06/21/93 17:00:45	25.07	0.010	8.05	7.54	174	0.000
15	06/21/93 17:15:45	26.50	0.010	7.88	7.56	174	0.323
16	06/21/93 17:30:45	27.00	0.010	7.82	7.58	172	0.369
17	06/21/93 17:45:45	27.07	0.010	7.80	7.60	169	0.069
18	06/21/93 18:00:45	26.81	0.010	7.84	7.60	167	0.115
19	06/21/93 18:15:45	26.50	0.010	7.87	7.60	165	0.115
20	06/21/93 18:30:45	26.19	0.010	7.92	7.59	164	0.115
21	06/21/93 18:45:45	25.80	0.010	7.95	7.59	161	0.115

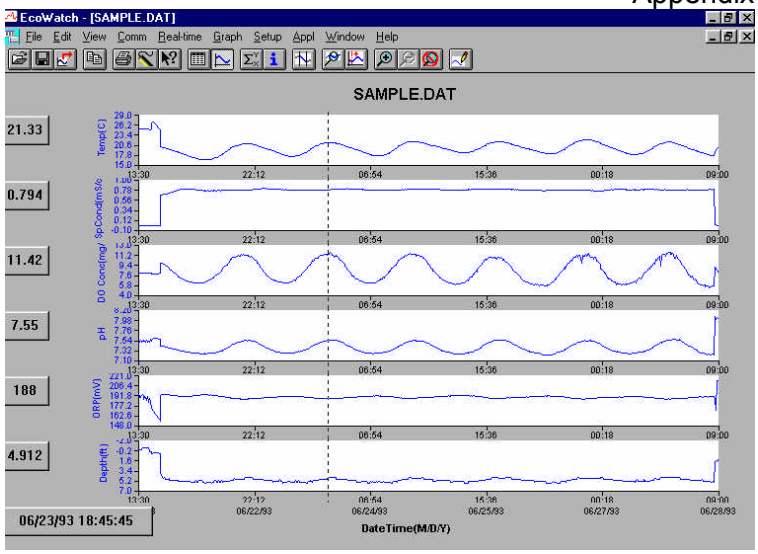
Now return to the original graphic display by toggling the **Table** button "off" and **Graph** button "on".



From the **Setup** menu, click **Graph**. Click **2 Traces per Graph** and notice that the parameters are now graphed in pairs for easy comparison of parameters.

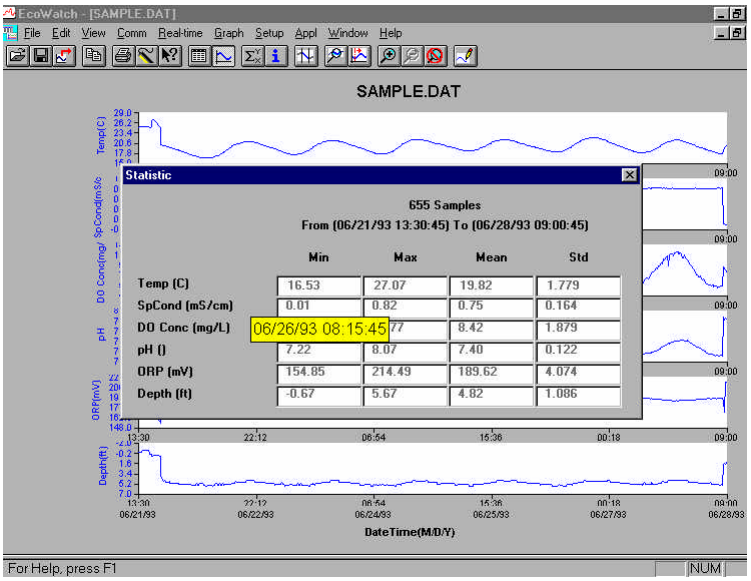


Click **1 Trace per Graph** to return the display to the original setting. Move the cursor to any position in the graph, then click and hold the right mouse button.



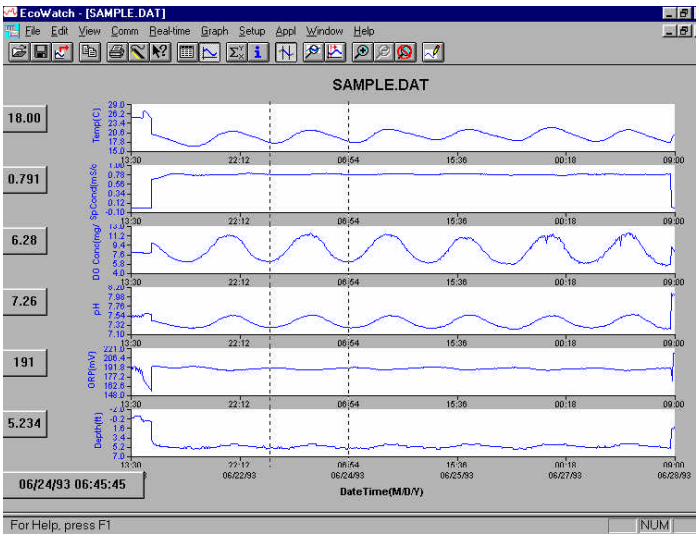
Note that the exact measurements for this point in time are displayed to the left of the graph. While holding down the right mouse button, move to another area on the graph. Notice how the measurements change as you move. When you release the mouse button, the display returns to normal.

To view statistical information for the study, click the **Statistics** button on the toolbar. On the statistics window, click on any min or max value to display the time when it occurred.

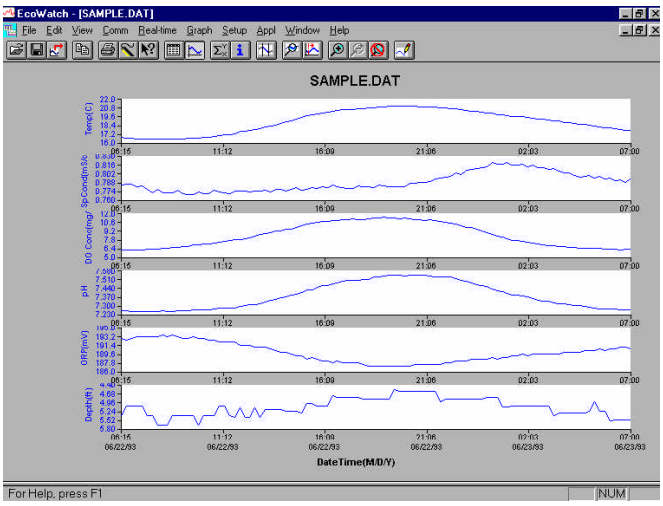


After viewing statistics, click the "x" at the upper right to close the window and return to the normal display.

Now click on the delimiter icon in the toolbar and then move the displayed icon to the graph. Click at the two points shown by dotted lines in the display below, being sure that the first click is to the left of the second.

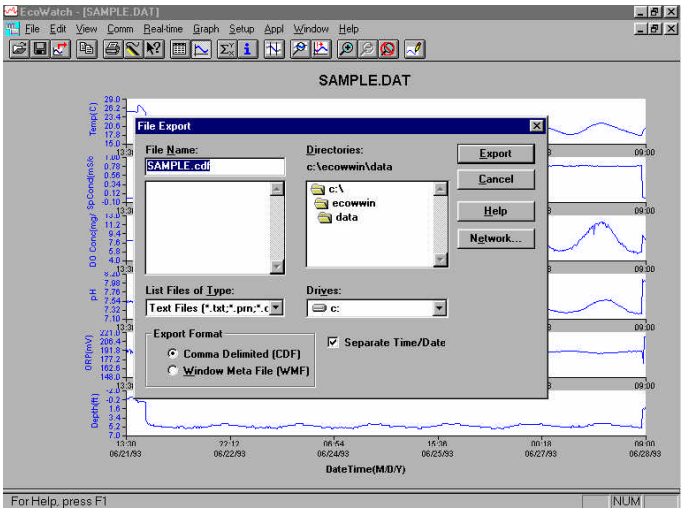


The data between the two selected points will then be graphed in higher resolution as shown below.

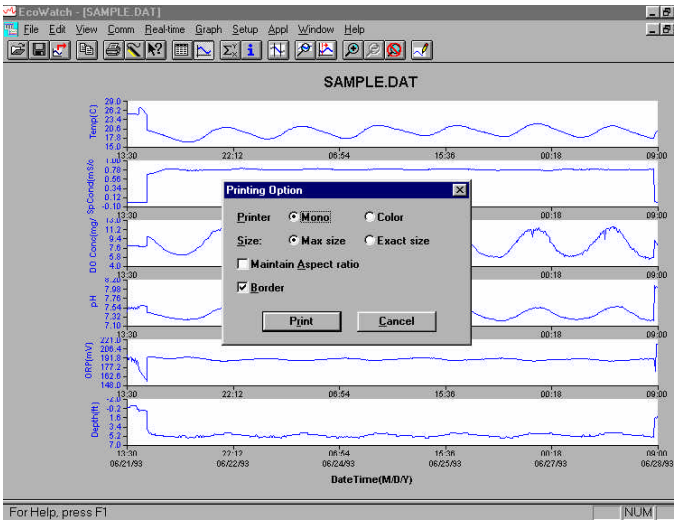


To return to the complete data set, select **Graph** from the toolbar and then click **Cancel Limits**.

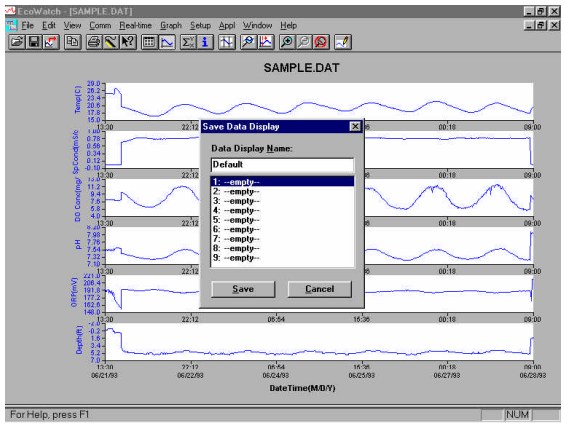
Now select the icon from the Toolbar to create a new data file which will allow your data to be imported into spreadsheets. Select the default export settings for a Comma Delimited File (.CDF) and click OK. A new spreadsheet-importable file (SAMPLE.CDF) is now present in the same folder as the SAMPLE.DAT file.



Now select the icon from the toolbar to print the plot. Accept the default settings and click OK to complete the printing operation.



Finally, end the tutorial by saving the **Data Display** in the format shown. From the **File** menu, click **Save Data Display**.



Then type “Default” for the file name and click **Save**. The parameters, colors, format, and x-axis time interval associated with the current display are now saved and can be accessed any time in the future. Nine different data displays may be saved for any data file. You can easily switch between various displays of the data. The data files can be accessed by clicking **Load Data Display** from the file menu and then selecting the desired presentation.

20.2.1 Summary of Toolbar Capability

The EcoWatch toolbar includes buttons for some of the most common commands in EcoWatch, such as **File Open**. To display or hide the toolbar, open the **View** menu and click on the **Toolbar** command. A check mark appears next to the menu item when the toolbar is displayed.

The toolbar is displayed across the top of the application window, below the menu bar.

Click To:

Open an existing data file (.DAT). EcoWatch displays the **Open** dialog box, in which you can locate and open the desired file.

Save the working Data Display of the active data file. EcoWatch displays the **Save Data Display** dialog box in which you can overwrite existing Data Display or save to a new one.

Export data as a graph in Window Meta File (.WMF) format or as data in Comma Delimited (.CDF) format.

Copy the whole graph page or data from the selection on the table to the clipboard.

Print the active graph page or table page depending on which one is currently active.

Open a new terminal window to communicate with the sonde.

Access context sensitive help (Shift+F1).

Toggle table window during file processing.

Toggle graph window during file processing.

Display study statistics.

Display study info.

Limit the data to be processed in a study.

Enlarge a selective portion of graph.

Center the graph under the cursor.

Enlarge graph or table 20%.

Reduce graph or table 20%.

Return graph or table to its normal state (unzoom)

Redraw the graph.

20.2.2 Other Capabilities

The above tutorial and function list for the toolbar provide basic information to allow you to view and analyze the field data which was stored in your 556 MPS. Some of the other commonly used capabilities of EcoWatch which the user may want to explore are listed below:

- Customize the units for each parameter, e.g., report uS/cm instead of mS/cm for conductivity.
- Customize the order of parameters in each plot or report.
- Customize the colors and fonts of each data display.
- Manually scale the y-axis sensitivity for each parameter.
- Merging of two or more data files with compatible parameter formats
- View information about the study such as number of points, instrument serial number, etc. which was stored in the 556 with the data.
- Print data reports in different statistical formats.
- Create plots of parameter vs. parameter rather than parameter vs. time.

These additional features of EcoWatch for Windows are explained in detail in the YSI 6-series manual (which can be downloaded at no cost from the YSI Web Site as described above) and the Help selection in the EcoWatch menubar. To purchase a hard copy of the 6-series manual, contact YSI Customer Service using the contact information in *Appendix E Customer Service*.

21.Appendix H Calibration Record Information

When your YSI 556 MPS sensors are initially calibrated, relevant information about the sensors will be stored in a separate file in the YSI 556 MPS memory.

NOTE: This file, by default, will have the name "556 Circuit Board Serial Number.glp." The circuit board serial number is assigned at the factory and has a hexadecimal format such as 000080A4. Thus the default calibration record file would be designated 00080A4.glp. Refer to Section *10.7 GLP Filename* to change the filename.

The information in the calibration record will track the sensor performance of your instrument and should be particularly useful for programs operating under Good Laboratory Practices (GLP) protocols.

21.1 Viewing the Calibration Record (.glp) File

NOTE: Make certain that you have performed a calibration on at least one of the sensors associated with your YSI 556 MPS.

1. Follow the procedures outlined in Section *8.3 View File*.

21.2 Uploading the Calibration Record (.glp) File

NOTE: Make certain that you have performed a calibration on at least one of the sensors associated with your YSI 556 MPS.

1. Follow the procedures outlined in Section *8.4 Upload to PC*.

21.3 Understanding the Calibration Record (.glp) File

1. Open a calibration record file. Refer to Section *8.3 View File*.

2. Use the arrow keys to scroll horizontally and/or vertically to view all the data.

00008003.glp

m/d/y	hh:mm:ss	S/N
01/24/2001	08:17:51	00008003
01/24/2001	08:17:51	00008003
01/24/2001	08:17:51	00008003
01/24/2001	08:17:51	00008003
01/24/2001	08:17:51	00008003
01/24/2001	08:17:51	00008003
01/24/2001	08:17:51	00008003
01/24/2001	08:17:51	00008003
01/24/2001	08:25:40	00008003
01/24/2001	08:25:40	00008003

735.9mmHg

01/24/2001 08:39:53

Figure 21.1 Calibration Record Screen 1

00008003.glp

Type	Value
Conductivity gain	1.000000
DO gain	1.000000
pH gain (pH-7)*K/mV	-5.05833
pH offest (pH-7)*K	0.000000
ORP offset mV	0.000000
TDS constant	0.650000
Barometer offset PSI	0.000000
DO gain	1.110250
pH gain (pH-7)*K/mV	-5.05833
pH offest (pH-7)*K	-12.2899

735.9mmHg

01/24/2001 08:39:19

Figure 21.2 Calibration Record Screen 2

NOTE: Each sensor (not parameter) is characterized by either 1 line (Conductivity, Dissolved Oxygen, ORP, TDS, or Barometer (Optional)) or 2 lines (pH) of calibration documentation.

The left hand portion of each calibration entry shows the date and time that a calibration of a particular sensor was performed. In addition, each calibration entry is characterized by the instrument serial number, as defined by YSI. See Figure 21.1 Calibration Record Screen 1. The right hand portion shows the YSI designation of the calibration constants and their values after their calibration has been performed. A more detailed description of the calibration constants is provided below:

- **Conductivity Gain** – A relative number which describes the sensitivity of the sensor. Basically, the value represents the calculated cell constant divided by the typical value of the cell constant (5 cm^{-1}).

- **DO Gain** – A relative number which describes the sensitivity of the sensor. Basically, the value represents the sensor current at the time of calibration divided by the typical value of the sensor current (15 uA).

- **pH Gain** – A number which basically represents the sensitivity of the pH sensor. To remove the effect of temperature on the slope of the relationship of probe output in mv versus pH, the value of pH/mv is multiplied by the temperature in degrees Kelvin (K).

- **pH Offset** – A number which basically represents the offset (or intercept) of the relationship of probe output in mv versus pH, the value of pH is multiplied by the temperature in degrees Kelvin (K).

Anytime you perform a calibration, information concerning the calibration constants will be logged to the Calibration Record file (.glp file). However, if the **Delete All Files** command is used, Refer to Section *8.6 Delete All Files*, the Calibration Record file will also be lost. It is critical that this file should be uploaded to your PC prior to issuing a **Delete All Files** command. Refer to Section *8.4 Upload to PC*.

YSI *incorporated*

YSI Environmental
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Hach® Portable Turbidimeter
Instrument and Procedure Manual

CAT. NO. 46500-88

PORTABLE TURBIDIMETER

Model 2100P

Instrument and Procedure Manual

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OPERATION

DANGER

Handling chemical samples, standards, and reagents can be dangerous. Review the necessary Material Safety Data Sheets and become familiar with all safety procedures before handling any chemicals.

DANGER

La manipulation des échantillons chimiques, étalons et réactifs peut être dangereuse. Lire les Fiches de Données de Sécurité des Produits (FDSP) et se familiariser avec toutes les procédures de sécurité avant de manipuler tous les produits chimiques.

PELIGRO

La manipulación de muestras químicas, estándares y reactivos puede ser peligrosa. Revise las fichas de seguridad de materiales y familiarícese con los procedimientos de seguridad antes de manipular productos químicos.

GEFAHR

Da das Arbeiten mit chemischen Proben, Standards und Reagenzien mit Gefahren verbunden ist, empfiehlt die Hach Company dem Benutzer dieser Produkte dringend, sich vor der Arbeit mit sicheren Verfahrensweisen und dem richtigen Gebrauch der Chemikalien vertraut zu machen und alle entsprechenden Materialsicherheitsdatenblätter aufmerksam zu lesen.

PERIGO

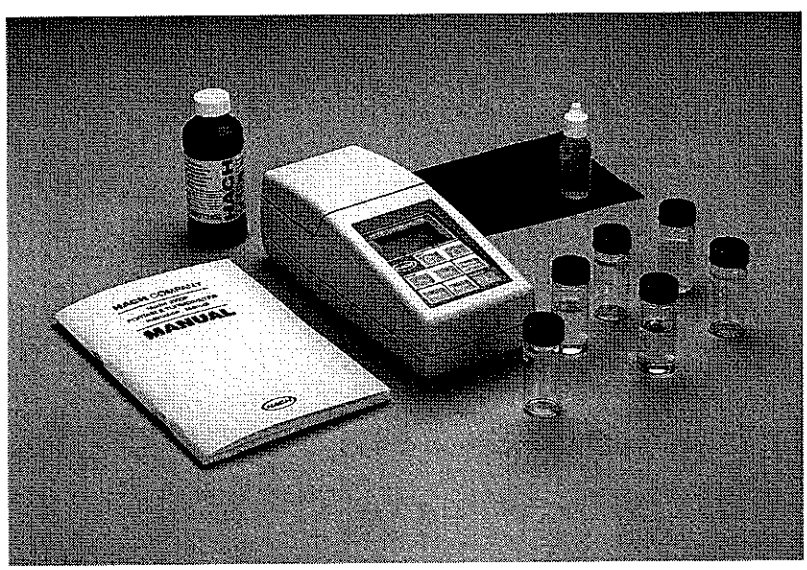
A manipulação de amostras, padrões e reagentes químicos pode ser perigosa. Reveja a folha dos dados de segurança do material e familiarize-se com todos os procedimentos de segurança antes de manipular quaisquer produtos químicos.

SECTION 1 DESCRIPTION

1.1 General Description

The Hach Model 2100P Portable Turbidimeter (*Figure 1*) measures turbidity from 0.01 to 1000 NTU in automatic range mode with automatic decimal point placement. The manual range mode measures turbidity in three ranges: 0.01 to 9.99, 10 to 99.9 and 100 to 1000 NTU. Designed primarily for field use, the microprocessor-based Model 2100P has the range, accuracy, and resolution of many laboratory instruments. The instrument operates on four AA batteries or with an optional battery eliminator. Rechargeable nickel-cadmium cells may be used, but cannot be recharged in the instrument. The instrument automatically shuts off after 5.5 minutes if no keystrokes occur (does not influence operation). If this occurs, simply turn the instrument on – the 2100P will resume operation as if the power had not been interrupted. The instrument, all standard accessories, and the optional battery eliminator may be conveniently stored in the carrying case.

Figure 1 2100P Turbidimeter and Accessories



Note: *Avoid prolonged exposure to ultraviolet light and sunlight.*

Note: *Do not hold the instrument during measurements; place the instrument on a flat, steady surface.*

1.2 Accessories

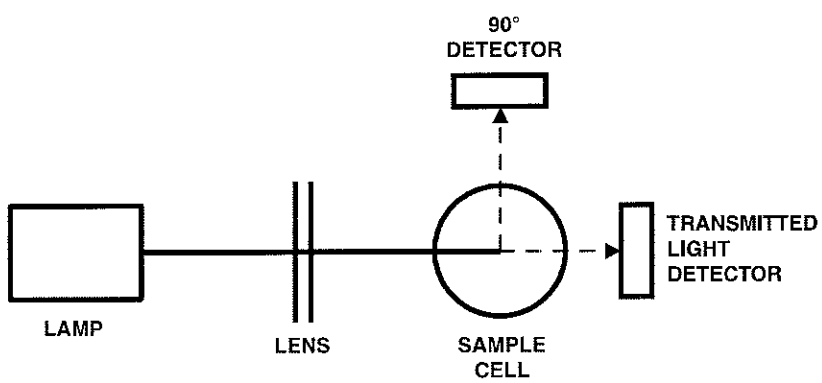
Accessories supplied with the turbidimeter include three sample cells; three Gelex® Secondary Standards; one 100 mL bottle each of: <0.1-NTU, 20-NTU, 100-NTU, and 800-NTU StablCal® Stabilized Formazin Standards; 4 AA alkaline batteries; 15 mL of silicone oil; oiling cloth; carrying case; instrument manual; and quick reference card

1.3 Principle of Operation

The Model 2100P Portable Turbidimeter operates on the nephelometric principle of turbidity measurement This instrument meets the design criteria specified by the United States Environmental Protection Agency, Method 180.1

The optical system* (*Figure 2*) includes a tungsten-filament lamp, a 90° detector to monitor scattered light and a transmitted light detector. The instrument's microprocessor calculates the ratio of the signals from the 90° and transmitted light detectors This ratio technique corrects for interferences from color and/or light absorbing materials (such as activated carbon) and compensates for fluctuations in lamp intensity, providing long-term calibration stability. The optical design also minimizes stray light, increasing measurement accuracy.

Figure 2 Ratio Optical System



* Patent number 4,198,161; other patents pending

1.4 Preparation for Use

1.4.1 Unpacking

Remove the instrument and accessories from the shipping box and inspect them for damage that may have occurred due to rough handling or extreme weather conditions. Verify the following are present:

- Model 2100P Portable Turbidimeter
- Instrument Manual (with quick reference card)
- StablCal Stabilized Formazin Standard, <0.1 NTU*, 100 mL
  StablCal Stabilized Formazin Standard, 20 NTU, 100 mL
  StablCal Stabilized Formazin Standard, 100 NTU, 100 mL
  StablCal Stabilized Formazin Standard, 800 NTU, 100 mL
- Standardization Kit containing Gelex Secondary Standards (0-10, 0-100 and 0-1000 ranges) plus nine sample cells with caps.
- Silicone Oil, 15-mL (0.5 oz) dropping bottle
- Oiling Cloth
- Carrying Case
- Four AA alkaline batteries

If any of the items are missing or damaged, please contact the Customer Service Department, Hach Company, Loveland, Colorado. The toll-free number in the United States is 800-227-4224. International customers should contact the Hach office or authorized distributor serving your area. Refer to *REPAIR SERVICE* on page *68*. **Please do not return the instrument without prior authorization from Hach.**

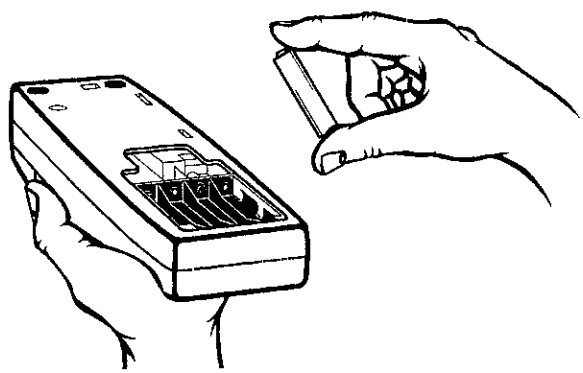
1.4.2 Battery Installation

The instrument is shipped completely assembled without the batteries installed. Before use, install the four AA alkaline batteries or connect the battery eliminator (*Figure 3*). For battery operation, remove the

* Used in place of the dilution water standard when performing a calibration.

battery compartment cover on the instrument bottom and install the batteries Correct battery polarity is shown on the battery holder The instrument will not function if the batteries are not installed correctly Reinstall the battery compartment cover

Figure 3 Battery Installation



1.4.3 Using the Battery Eliminator and Rechargeable Batteries

For operation with the optional battery eliminator, plug the eliminator jack into the connector on the turbidimeter side. The battery eliminator may be used with or without the batteries installed. **The eliminator will not charge batteries.** Rechargeable batteries may be used in the instrument, but must be removed for recharging. See *HOW TO ORDER* on page *67* for ordering information. To prolong battery life, the instrument lamp turns on temporarily when the **READ** key is depressed Batteries are not necessary for battery eliminator operation.

1.4.4 Calibration

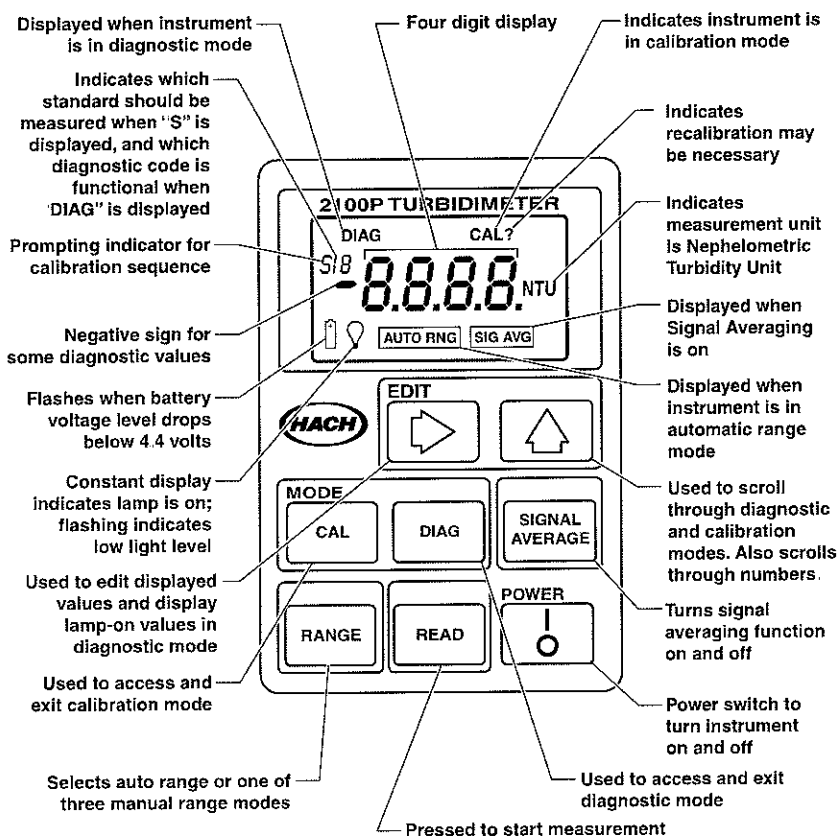
The 2100P Portable Turbidimeter is calibrated with Formazin Primary Standard at the factory and **does not require recalibration before use.** Hach recommends recalibration with formazin once every three months, or more often as experience dictates The Gelex Secondary Standards supplied with the instrument are labelled with general ranges for application, but must be assigned values before use from formazin calibration. See *Section 3.6* on page *27* for calibration instructions.

SECTION 2 TURBIDITY MEASUREMENT

2.1 Operating Controls and Indicators

Figure 4 shows the 2100P controls and indicators. Refer to *SECTION 3* for a detailed description of each control and indicator

Figure 4 Keyboard and Display with Descriptions



2.2 Turbidity Measurement

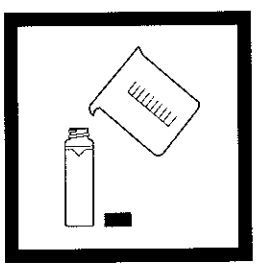
Measurements may be made with the signal average mode on or off and in manual or automatic range selection mode. Using automatic range selection is recommended. Signal averaging uses more power and should be used only when the sample causes an unstable reading. Signal averaging measures and averages ten measurements while displaying

intermediate results The initial value is displayed after about 11 seconds and the display is updated every 1.2 seconds until all ten measurements are taken (about 20 seconds). After this, the lamp turns off, but the final measured turbidity value continues to be displayed until another key is pressed

When not in signal average mode, the final value is displayed after about 13 seconds

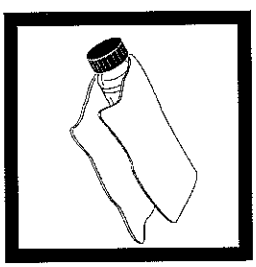
Accurate turbidity measurement depends on good measurement technique by the analyst, such as using clean sample cells in good condition and removing air bubbles (degassing). Refer to *Section 2.3* on page *10* for a detailed discussion of measurement techniques

2.2.1 Turbidity Measurement Procedure

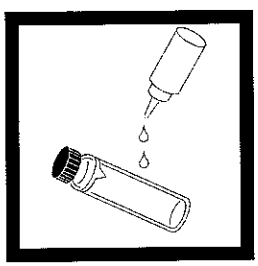


1. Collect a representative sample in a clean container. Fill a sample cell to the line (about 15 mL), taking care to handle the sample cell by the top. Cap the cell. (See *Section 2.3* on page *10* for more information about collecting a representative sample).

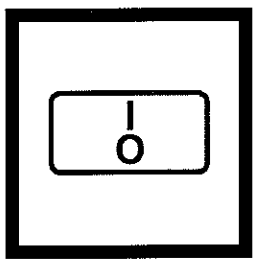
Note: *The instrument automatically shuts off after 5.5 minutes if no keystrokes occur. To resume operation, press* **I/O**.



2. Wipe the cell with a soft, lint-free cloth to remove water spots and fingerprints.

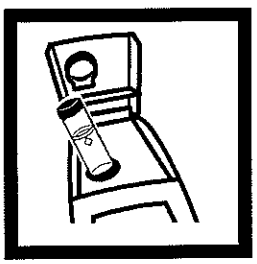


3. Apply a thin film of silicone oil. Wipe with a soft cloth to obtain an even film over the entire surface

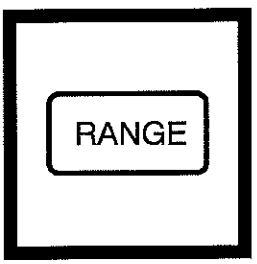


4. Press: **I/O**.

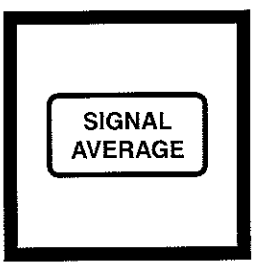
The instrument will turn on Place the instrument on a flat, sturdy surface Do not hold the instrument while making measurements.



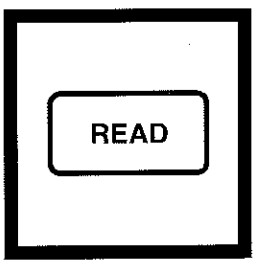
5. Insert the sample cell in the instrument cell compartment so the diamond or orientation mark aligns with the raised orientation mark in front of the cell compartment.
Close the lid.



6. Select manual or automatic range selection by pressing the **RANGE** key. The display will show **AUTO RNG** when the instrument is in automatic range selection.



7. Select signal averaging mode by pressing the **SIGNAL AVERAGE** key. The display will show **SIG AVG** when the instrument is using signal averaging Use signal average mode if the sample causes a noisy signal (display changes constantly)



8. Press: **READ**

The display will show **- - - - NTU**, then the turbidity in NTU. Record the turbidity after the lamp symbol turns off.

Note: *The instrument defaults to the last operating mode selected If automatic range mode and signal averaging were used on the previous measurements, these options will automatically be selected for subsequent samples.*

2.2.2 Measurement Notes

- Always cap the sample cell to prevent spillage of sample into the instrument.
- When taking a reading, place the instrument on a level, stationary surface. It should not be held in the hand during measurement.
- Always close the sample compartment lid during measurement and storage.
- Always use clean sample cells in good condition. Dirty, scratched, or damaged cells can cause inaccurate readings.
- Do not leave a sample cell in the cell compartment for extended periods of time. This may compress the spring in the cell holder.
- Remove sample cell and batteries from instrument if the instrument is stored for extended time period (more than a month).
- Avoid operating in direct sunlight
- Make certain cold samples do not "fog" the sample cell.
- Avoid settling of sample prior to measurement.
- Keep sample compartment lid closed to prevent dust and dirt from entering

2.3 Measurement Techniques

Proper measurement techniques are important in minimizing the effects of instrument variation, stray light and air bubbles. Regardless of the instrument used, measurements are more accurate, precise and repeatable if the analyst pays close attention to proper measurement techniques

Measure samples immediately to prevent temperature changes and settling. Avoid sample dilution when possible. Particles suspended in the original sample may dissolve or otherwise change characteristics when the sample temperature changes or when the sample is diluted, resulting in a non-representative sample measurement.

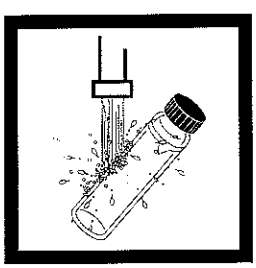
2.3.1 Cleaning Sample Cells

Cells must be extremely clean and free from significant scratches. The glass used to make cells is easily scratched – manufacturing cells free of minor scratches and other imperfections is difficult. However, minor imperfections are effectively masked by applying silicone oil as outlined in *Section 2.3.2*

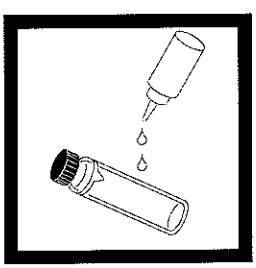
Clean the inside and outside of the cells by washing with laboratory detergent. Follow with multiple rinses of distilled or deionized water. Allow cells to air dry. Handle cells only by the top to minimize dirt, scratches and fingerprints in the light path

2.3.2 Oiling the Sample Cell

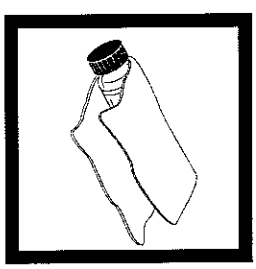
Applying a thin coat of silicone oil will mask minor imperfections and scratches which may contribute to turbidity or stray light. Use silicone oil equivalent to Hach Cat. No. 1269-36. This silicone oil has the same refractive index as glass. When applied in a thin, uniform coat, the oil fills in and masks minor scratches and other imperfections in the glass. Apply the oil uniformly by wiping with a soft, lint-free cloth
Avoid application of excess oil. Applying excess oil may retain dirt and contaminate the instrument's cell compartment



1. Thoroughly clean the sample cell



2. Apply a small bead of silicone oil from the top to the bottom of the cell-- just enough to coat the cell with a thin layer of oil.



3. Using a soft, lint-free cloth, spread the oil uniformly, then wipe off the excess so that only a thin coat of oil is left. The cell should appear nearly dry with little or no visible oil

Note: *Soft, lint-free cloth (velvet) works well for oiling Store the oiling cloth with the sample cells and keep it free of dirt After a few applications of oil, the cloth will contain enough residual oil that simply wiping the cell with the oiled cloth will provide a sufficient oil coat on the sample cell. Periodically, add a small amount of oil to the sample cell surface to replenish the oil in the cloth.*

Note: *Only a thin coat of oil on the sample cells is necessary.* ***Avoid using excessive amounts of oil.***

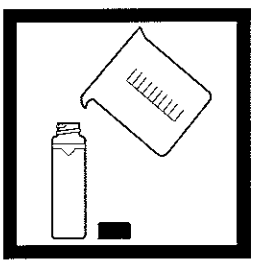
2.3.3 Orienting Sample Cells

Note: *When orienting and matching cells, it may be more efficient to use the continuous reading mode. The instrument performs continuous readings if the* **READ** *key is pressed and held. As long as the key is held, the lamp remains on and the display is updated every 1 2 seconds. The instrument cannot be used in continuous read mode if the Signal Averaging mode is on.*

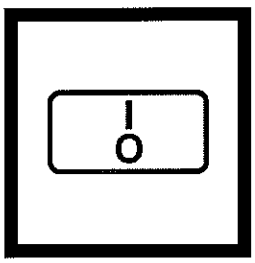
Precise measurements for very low turbidity samples require using a single cell for all measurements or optically matching the cells. Using one cell provides the best precision and repeatability When one cell is used, an orientation mark (other than the factory-placed diamond) can be placed on the cell so it's inserted into the instrument with the same orientation each time.

2.3.3.1 Orienting a single cell

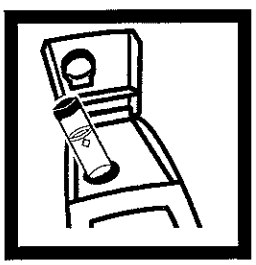
When using a single cell, make an index or orientation mark on the cell as follows:



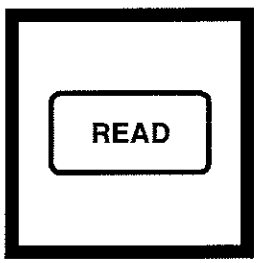
1. Fill the clean sample cell to the line with high quality water (< 0.5 NTU). Cap and wipe with lint-free cloth. Apply silicone oil. See *Section 3.6.2.2* on page *30* for more information about high quality water



2. Press: **I/O** to turn the instrument on.



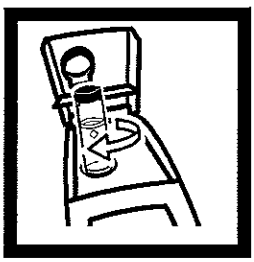
3. Insert the sample cell into the sample compartment. Close the cover



4. Press: **READ**

Record the cell's position in the cell compartment and the displayed reading

Note: *This procedure may be easier if the user holds the* **READ** *key through the whole process. This allows the lamp to remain on and make continuous readings.*

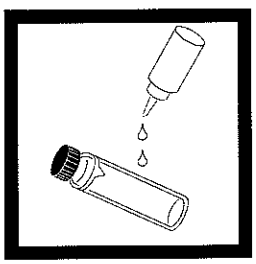


5. Remove the cell, rotate it slightly and reinsert it into the cell compartment. Close the cover, then press **READ**. Record the cell's position and the displayed reading.

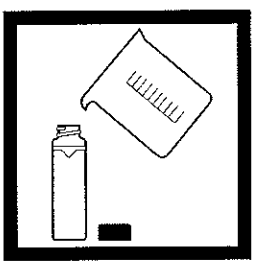
6. Repeat *step 5* until the lowest reading is displayed. Place an orientation mark on the cell's marking band near the top of the cell so the cell can be consistently inserted in the position that yields the lowest reading. When using the cell, always place it in the instrument so the orientation mark aligns with the raised mark on the instrument.

2.3.4 Matching multiple sample cells

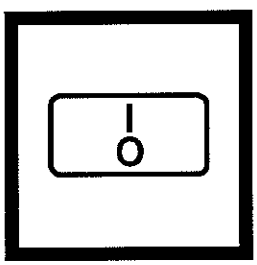
Precise measurements of very low turbidity samples require the cells be optically matched or a single cell be used for all measurements. If more than one cell is used, follow this procedure to match (index) the cells:



1. Clean and oil the sample cells as instructed in *Section 2.3.1* on page *11* and *Section 2.3.2* on page *11*.

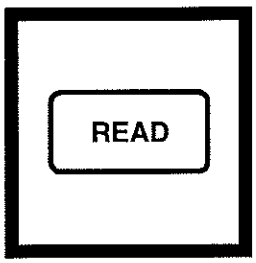


2. Fill the clean sample cells to the line with the same sample



3. Press: **I/O** to turn the instrument on.

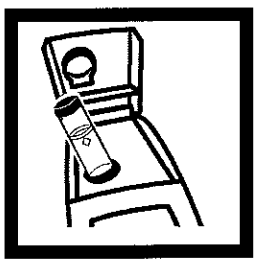
4. Insert the **first** sample cell into the sample compartment and close the cover.



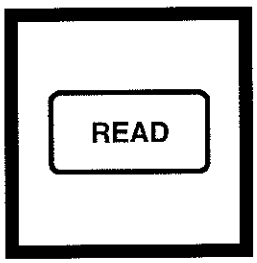
5. Press: **READ**

Record the cell's position in the cell compartment and the displayed reading. Place an orientation mark on the cell's marking band.

Note: *This procedure may be easier if the user holds the* **READ** *key through the whole process. This allows the lamp to remain on and make continuous readings.*



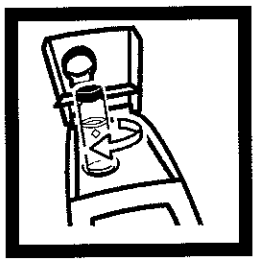
6. Insert the **second** sample cell into the cell compartment and close the cover.



7. Press: **READ**

Record the cell's position in the cell compartment and the displayed reading.

8. Remove the cell, rotate it slightly and reinsert into the cell compartment. Close the cover, then press **READ** again. Record the cell's position and the displayed reading.



9. Repeat *step 8* until the value displayed for the second cell is within 0.01 NTU (or 1%) of the value obtained for the first cell. Place an orientation mark on the second cell's marking band so it is consistently inserted in this position

Note: *Due to variability in glass, it may not be possible to match all cells*

10. Repeat *step 6* through *step 9* if matching other sample cells.

2.3.5 Removing Bubbles (Degassing)

Before measurement, removing air and other trapped gasses from the sample is strongly recommended, even if bubbles are not visible. Four degassing methods are commonly used:

1. applying a partial vacuum
2. adding a surfactant
3. using an ultrasonic bath
4. heating the sample

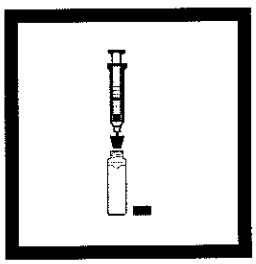
In some cases, more than one method may be necessary for effective bubble removal. For example, use of both a surfactant and ultrasonic bath may be necessary for some severe conditions. Use care with these techniques. If misused, sample turbidity can be altered.

Removing air bubbles by letting the sample stand for a period of time is not recommended. Particulates that cause turbidity may settle and the sample temperature may change. Both conditions may alter sample turbidity, resulting in measurements not representative of the original turbidity.

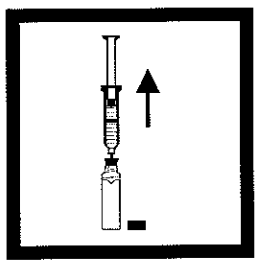
2.3.5.1 Application of vacuum

Apply a vacuum with any convenient, clean, oil-free vacuum source. The vacuum lowers the atmospheric pressure, allowing trapped bubbles to escape into the air above the sample. Vacuum works well with non-viscous samples (such as water) that don't contain volatile components. Applying vacuum to viscous, volatile-containing samples (paint resins) may cause the volatile components to come out of solution and aggravate the bubble problem.

To apply a vacuum, use a sample degassing kit equivalent to Cat No. 43975-00 (Degassing Kit) or 43975-10 (Degassing and Filtration Kit). These kits contain a syringe and rubber stopper for vacuum degassing. An electric or hand-operated pump equivalent to Cat No. 14283-00 or 14697-00, respectively, may also be used.



1. Fill a sample cell to the mark with sample Insert a #2 single-hole rubber stopper and syringe into the cell. If using a pump, insert a piece of glass tubing into the stopper



2. **Slowly** apply the vacuum by carefully pulling the plunger upward, then holding it. If using a hand or electric pump, connect the tubing to the vacuum pump with vacuum hose. Apply vacuum until visible gas bubbles disappear. **Slowly** release the vacuum. Remove the vacuum apparatus and cap the cell.

2.3.5.2 Adding a surfactant

Surfactants should be limited to severe problems when other degassing methods are ineffective. Surfactants change the surface tension of the water, which releases trapped gases. Hach recommends a surfactant such as Triton X-100 or the equivalent, Hach Cat No. 14096-37. Put one drop of Triton X-100 in the sample cell before adding sample.

Note: *Any turbidity contributed by surfactant addition is negligible*

This technique is very effective when the water is super-saturated with air. However, changing the surface tension may accelerate settling of turbidity-causing particles. Mix the sample gently, but thoroughly, and analyze as soon as possible after adding the surfactant. Avoid vigorous mixing as the surfactant may foam. Rinse the sample cells thoroughly between samples to prevent surfactant accumulation.

2.3.5.3 Using an ultrasonic bath

Note: *The time necessary to expel bubbles may vary from a few seconds to a minute or more. To avoid excessive application of ultrasound, a simple procedure can be followed. First, apply ultrasound until all visible bubbles are absent. Then measure the sample turbidity. Apply ultrasound for a short time period and again measure turbidity. Continue for several repetitions, noting the treatment time and turbidity readings. If turbidity begins to increase instead of decrease, the ultrasound waves have probably started to alter the suspended particles. Note the time it takes for this to occur and record it as the maximum time limit for ultrasonic treatment.*

Ultrasonic baths effectively remove gas bubbles from most samples, especially viscous liquids. However, the ultrasonic waves which cause degassing may also alter the characteristics of the particles causing the turbidity. Turbidity depends on the size, shape, composition and refractive index of the suspended particles. Excessive ultrasound application may alter particle size and shape, thus changing sample turbidity. In some cases, ultrasound may aggravate air bubble removal by fracturing the bubbles, making degassing more difficult.

1. Fill a clean sample cell to the line with sample. Leave uncapped.
2. Immerse the cell (1/2 to 2/3 immersed) in an ultrasonic bath and allow it to stand until visible bubbles are expelled.
3. Remove the cell, cap, then thoroughly dry the cell. Apply silicone oil as directed.

2.3.5.4 Application of heat

Whenever possible, avoid using heat to degas samples because heat may change the characteristics of the suspended particles and cause volatile components to come out of solution. Gentle heating may be helpful for degassing some very viscous samples when combined with application of vacuum or ultrasound. If heat is necessary, heat the sample only until degassing occurs. The simplest technique is to prepare a warm water bath and partially immerse the filled sample cell. Use the shortest time necessary for expelling visible bubbles. Cool sample to original sample temperature before taking measurements.

2.3.6 Measuring Overrange Samples

Nephelometric turbidity measurement depends on detection of light scattered from particles suspended in the liquid. If the turbidity is very high, a significant amount of light is blocked or absorbed by the particles and only a small amount of light reaches the detector. This results in a negative interference – the measured turbidity is lower than the actual turbidity. This condition is called "going blind". A multidetector ratioing instrument, such as the Hach 2100P Turbidimeter, minimizes this effect and extends the instrument range. Highly turbid samples may also be diluted, but this should be avoided when possible since it may alter the characteristics of the suspended particles and produce erroneous results.

Light absorbing particles such as activated carbon and highly colored samples may also cause an instrument to "go blind" Dilution may not correct for these interferences A ratioing instrument will correct for the presence of light absorbing particles and color

2.3.7 Condensation (fogging)

Condensation may occur on the outside of the sample cell when measuring a cold sample in a warm, humid environment Condensation interferes with turbidity measurement, so all moisture must be thoroughly wiped off the sample cell before measurement. If fogging recurs, let the sample warm slightly by standing at room temperature or immersing it in a warm bath for a short period. After warming, mix the sample thoroughly before measurement. Allowing samples to warm can alter sample turbidity, so it is best to avoid warming samples before measurement when possible

2.3.8 Calibration

Turbidimeters must be properly calibrated with a primary standard Hach recommends formazin or StablCal Stabilized Formazin for calibration. For U.S Environmental Protection Agency (USEPA) reporting, calibrate at least as often as required by the appropriate regulatory agencies. The frequency of calibration depends on environmental conditions (humidity, temperature) and use. If necessary, calibrate more frequently

Use secondary standards for periodic calibration checks. Please note that Gelex® standards must be assigned values after StablCal Stabilized Formazin calibration or formazin calibration and before use as secondary standards. Gelex standards must be recalibrated (values assigned) each time the instrument is calibrated with StablCal Stabilized Formazin or formazin. See *Section 3.6* on page *27* for detailed information on the use of StablCal Stabilized Formazin, formazin, and Gelex standards.

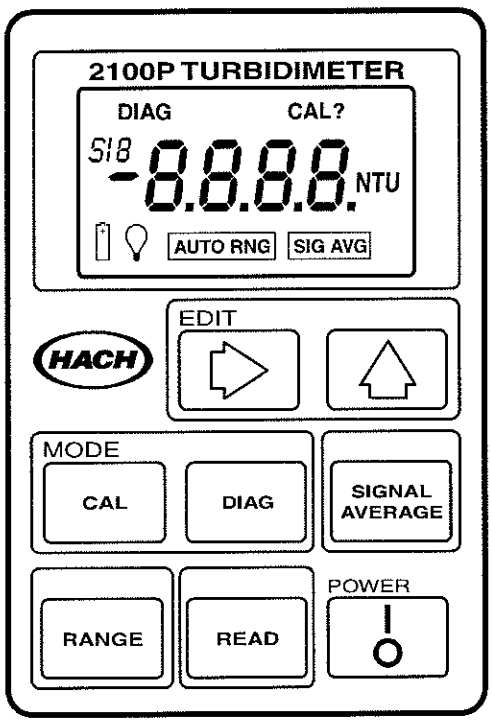
2.3.9 Representative Sampling

A representative sample accurately reflects the true condition of the water source from which the sample was taken. To ensure a representative sample, gently, but thoroughly, mix every sample before aliquots are taken. Do not allow the sample to settle.

When sampling from a tap in a distribution system or treatment plant, allow the water to run for at least five minutes before sampling. When sampling from a stream, reservoir, clarifier, or storage tank, collect at least one liter (1 quart) and thoroughly mix before measurement. If the water source is not uniform, it may be necessary to sample several locations at varying depths and combine the samples into a single, well-mixed composite sample before measurement.

SECTION 3 OPERATION

3.1 Operational Controls and Indicators



Key	Description
I O	Power key to turn instrument on and off. If no keys are pressed for 5.5 minutes, the instrument turns off automatically.
READ	Depressed to perform a measurement. To conserve battery power, the lamp turns on only when **READ** is depressed A reading is displayed about 12 seconds after the key is depressed. During the delay, a flashing **NTU** is displayed. After the reading is displayed, the lamp turns off and the reading continues to be displayed. Continuous readings may be done by holding this key if **not** in the Signal Averaging mode. After the initial delay, the reading is updated every 1.2 seconds.
CAL	Used to perform a calibration or review calibration data Also terminates a calibration or calibration review and returns to the 2100P measurement mode.

Key	Description
△	Edits a flashing digit in the calibration mode or sequences through the calibration standards (S0,S1, S2, S3) or diagnostic menu.
▷	Used to move the editing cursor to the digits being edited in the calibration mode or initiate editing of a standard value
SIGNAL AVERAGE	Turns the signal averaging function on or off
DIAG	Selects the diagnostic mode.
RANGE	Selects Auto Range or Manual Range (one of three manual modes).

Display Icon	Description
DIAG	Turns on after the **DIAG** is pressed to access the diagnostic mode A number displayed under the **DIAG** icon (1-9) indicates which diagnostic function is active. See *Section 5 1* on page *57* for more information on diagnostic codes.
CAL	Turns on after the **CAL** key is pressed to access the calibration mode and remains on during the calibration.
CAL?	Appears after calibration if a value entered during calibration is outside an acceptable range. May indicate an operator error or possible instrument malfunction. Flashing **CAL?** indicates the default calibration coefficients are being used (even after a user-calibration has been done) or that no calibration data is currently stored.
S__	Displayed during calibration The S is followed by a number to indicate which standard value is currently being edited or displayed. Flashing number is prompting user for measurement of **S0**, **S1**, **S2** or **S3** to establish a calibration. Steady number identifies which standard's value is being displayed.
	Flashes when the battery voltage drops to 4 4 volts as an indication to change batteries At <4 0 volts, the instrument automatically shuts off.
	The lamp symbol is constantly on when the lamp is on and flashes after a reading if a marginal light level reaches the transmitted light detector. A flashing icon indicates the sample may be too turbid (not within measurement range) and needs dilution or the lamp needs replacing

Display Icon	Description
SIGNAL AVERAGE	Indicates the signal averaging mode is on The icon turns off if signal averaging is not selected.
AUTO RNG	Indicates instrument is in automatic range mode. The icon turns off when manual range mode is selected.
8888	The 4-digit display is active when the instrument is on (measurements are displayed to three digits). After the **READ** key is pressed - - - - is displayed during wait periods.
NTU	Identifies the measurement units- Nephelometric Turbidity Units. This icon is active during measurements and in the calibration mode.

3.2 Using the Read Key

To preserve battery power and prolong lamp life, the lamp turns on only after the **READ** key is pressed. Pressing the key turns the instrument lamp on; after about 12 seconds, the lamp turns off, but the measurement value continues to be displayed. After the first measurement, a four-second recovery time occurs before another measurement can be started. If **READ** is pressed during the recovery time, the display will begin flashing, but the lamp will not turn on until the full four seconds have passed. If no other key strokes occur within 5.5 minutes, the instrument turns off.

3.2.1 Continuous Reading

The instrument cannot be used in continuous read mode if the Signal Averaging mode is on

The instrument will perform continuous readings if the **READ** key is pressed and held. As long as the key is held, the lamp remains on and the display is updated every 1 2 seconds.

3.3 Using the Signal Averaging Key

The signal averaging mode compensates for reading fluctuations caused by drifting of sample particles through the light path Signal averaging is turned on or off by pressing the **SIGNAL AVERAGE** key The **SIG AVG** icon is displayed when signal averaging is on.

Signal averaging measures and averages ten measurements while displaying intermediate results The initial value is displayed after about

11 seconds and the display is updated every 1.2 seconds until all ten measurements are taken (about 22 seconds). After 22 seconds, the lamp turns off, but the final measured turbidity value continues to be displayed until another key is pressed.

When signal averaging is off, the instrument takes three measurements, the microprocessor averages them, then displays the average. If the **READ** key is held during measurement, the initial value is displayed in 12 seconds and is updated every 1.2 seconds as long as the **READ** key is held.

When the instrument is turned on, the instrument defaults to the signal averaging mode which was used during the last measurement.

3.4 Using the Range Selection Key

As shipped, the instrument defaults to automatic range mode. The first time the **RANGE** key is pressed, the instrument goes into manual range mode. The second, third, and fourth key strokes put the instrument in the 0.00-9.99, 10 to 99.9 or 100-1000 NTU range, respectively. Another key stroke brings the selection back to automatic range mode. When the automatic range mode is selected, the **AUTO RNG** icon is displayed. Range selection can be done any time except when a measurement or calibration is in progress.

When the instrument is turned on, the instrument defaults to the range mode and measurement range which was used during the last measurement.

3.5 Restoring the Default Calibration

To restore and use the default calibration, turn the instrument off. Press and hold **DIAG**, then press and release **I/O**. Release **DIAG** when the software version number disappears from the display. (For models with serial number less than 920300000800, **2100** disappears.) This clears any user-entered calibration from memory; the 2100P will use the default calibration for measurement. **CAL?** will appear and continue to flash until a user-entered calibration is successfully completed.

For best results, a user-entered calibration should be done every three months.

3.6 Calibration

Calibration of the 2100P Turbidimeter is based on formazin, the primary standard for turbidity. The instrument's electronic and optical design provide long-term stability and minimize the need for frequent calibration. The two-detector ratioing system compensates for most fluctuations in lamp output. **A formazin recalibration should be performed at least once every three months,** more often if experience indicates the need. When calibration is necessary, use a primary standard such as StablCal™ Stabilized Standards or formazin standards.

Hach Company only recommends the use of StablCal® Stabilized Formazin or formazin standards for the calibration of Hach turbidimeters. Hach Company cannot guarantee the performance of the turbidimeter if calibrated with co-polymer styrene divinylbenzene beads or other suspensions.

3.6.1 StablCal Stabilized Formazin Standards*

Most consistent results will be achieved with the use of StablCal Stabilized Formazin Standards for calibration. Refer to *Section 3.6.1.2* and *Section 3.6.1.3* for information on preparing the standards for use.

Note: *Hach StablCal Stabilized Formazin in 20-, 100-, and 800-NTU values is packaged in convenient sets for calibration of the 2100P Turbidimeter. The set may be ordered in 500-mL size bottles by specifying Cat. No. 26594-00, in 100-mL size bottles by specifying Cat. No. 26594-10 or in sealed vials by ordering Cat. No. 26594-05. (See OPTIONAL ACCESSORIES AND REAGENTS on page 61.)*

3.6.1.1 Storing and Handling StablCal Stabilized Standards

For optimum results when using StablCal Stabilized Standards, adhere to the following recommendations:

- Do not transfer the standard to another container for storage.
- Do not return standard from the sample cell back into the its original container. Standard contamination will result.
- Store standards between 0 and 25 °C

* StablCal Stabilized Formazin is cited as a primary standard in Hach Method 8195, an acceptable version of USEPA Method 180.1

- For long-term storage, refrigeration at 5 °c is recommended. Do not store above 25 °C.
- Allow the standard to acclimate to ambient instrument conditions before use (not to exceed 40 °C).
- Store away from direct sunlight. Store vials in their respective kit or shipping box with the cover in place

3.6.1.2 Preparing Bulk StablCal Stabilized Standards

Bulk standards that have been sitting undisturbed for longer than a month must be shaken to break the condensed suspension into its original particle size. Start at *step 1* for these standards. If the standards are used on at least a weekly interval, start at *step 3*.

Important Note: These instructions do not apply to <0.1-NTU* StablCal Standards; <0.1NTU StablCal Standards should not be shaken or inverted.

1. Shake the standard vigorously for 2-3 minutes to resuspend any particles
2. Allow the standard to stand undisturbed for 5 minutes
3. Gently invert the bottle of StablCal 5 to 7 times.
4. Prepare the sample cell for measurement using traditional preparation techniques. This usually consists of oiling the sample cell (see*Section 2.3.2* on page *11*) and marking the cell to maintain the same orientation in the sample cell compartment (see *Section 2.3.3* on page *12*). This step will eliminate any optical variations in the sample cell
5. Rinse the sample cell at least one time with the standard and discard the rinse.
6. Immediately fill the sample cell with the standard. Cap the sample cell and let it stand for one minute. The standard is now ready for use in the calibration procedure, *Section 3.6.3*.

* Used in place of the dilution water standard when performing a calibration

3.6.1.3 Preparing StablCal Stabilized Standards in Sealed Vials

Sealed vials that have been sitting undisturbed for longer than a month must be shaken to break the condensed suspension into its original particle size. Start at *step 1* for these standards. If the standards are used on at least a weekly interval, start at *step 3*

Important Note: These instructions do not apply to <0.1-NTU* StablCal Standards; <0.1NTU StablCal Standards should not be shaken or inverted.

1. Shake the standard vigorously for 2-3 minutes to resuspend any particles.

2. Allow the standard to stand undisturbed for 5 minutes.

3. Gently invert the vial of StablCal 5 to 7 times.

4. Prepare the vial for measurement using traditional preparation techniques. This usually consists of oiling the vial (see *Section 2.3.2* on page *11*) and marking the vial to maintain the same orientation in the sample cell compartment (see *Section 2.3.3* on page *12*) This step will eliminate any optical variations in the sample vial

5. Let the vial stand for one minute. The standard is now ready for use in the calibration procedure, *Section 3.6.3*.

3.6.2 Formazin Primary Standards

Perform the procedure in *Section 3.6.2.1* to prepare a 4000-NTU standard Alternately, order a 4000-NTU stock solution from Hach by specifying Cat. 2461-49 Prepare the dilutions from the 4000-NTU stock solution by following the instructions in *Section 3.6.2.4*

3.6.2.1 Preparing Formazin Stock Solution

Dilute formazin standard solutions from a 4000 NTU stock solution equivalent to Hach Cat. No 2461-49. The prepared stock solution is stable for up to one year when properly prepared. An alternative to purchasing the 4000 NTU stock solution is preparing a stock solution as follows:

1. Dissolve 5.000 grams of reagent grade hydrazine sulfate ($N_2H_4 \cdot H_2SO_4$) in 400 mL of distilled water
2. Dissolve 50.000 grams of pure hexamethylenetetramine in 400 mL of distilled water
3. Pour the two solutions into a 1000-mL volumetric flask and dilute to the mark with distilled water.
4. Let the solution stand for 48 hours at 25 °C (77 °F) to develop the 4000-NTU stock suspension. The standing temperature is critical for correct formation of formazin polymers
5. Mix the 4000 NTU suspension for at least ten minutes before use. Then it can be diluted with distilled or demineralized water to achieve a solution of the desired NTU value

Instead of diluting a formazin stock solution, StablCal Stabilized Formazin Standards may be used. Order the StablCal Calibration Set for the 2100P Turbidimeter, Cat.No. 26594-00 (500-mL bottles), Cat No 26594-10 (100 mL bottles), or Cat No. 26594-05 (sealed vials). (See *OPTIONAL ACCESSORIES AND REAGENTS* on page *61*.)

3.6.2.2 Correcting for Turbidity of Dilution Water

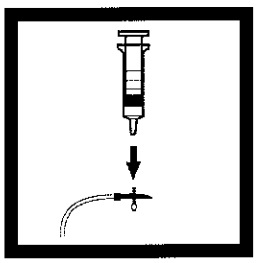
The 2100P Turbidimeter automatically compensates for turbidity contributed by dilution water when calculating the true value of the lowest formazin standard. Use high quality distilled or deionized water less than 0.5 NTU. The instrument will display E 1 after calibration if the dilution water turbidity is greater than 0 5 NTU. In this case, prepare the water as directed below

The value of the dilution water can be arbitrarily forced to zero (see calibration procedure). This is not recommended for most applications and, if done, should be done only if the dilution water turbidity is less than 0.2 NTU

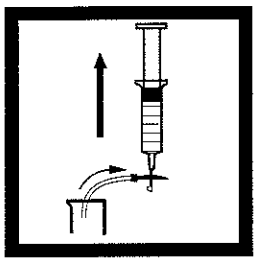
3.6.2.3 Preparing Dilution Water

Note: *Use the same dilution water for all dilutions and the sample blank.*

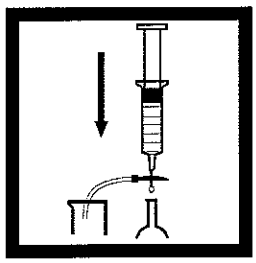
Collect at least 1000 mL of high quality dilution water (distilled or deionized water) The 2100P Turbidimeter, as received from the factory, is precalibrated and may be used to check the dilution water turbidity If the turbidity is greater than 0.5 NTU, filter the water with the Sample Filtration and Degassing Kit (Cat. No. 43975-10) or equivalent When measuring low range turbidity, clean all glassware with 1:1 hydrochloric acid and rinse several times with dilution water. If the glassware is not used immediately, use stoppers to prevent contamination from small particles



1. Attach the syringe to the 3-way valve by gently twisting the square end into the syringe tip. Attach the connector, tubing and a 0.2 micron filter (clear part faces syringe) as shown. Be sure the connections are tight



2. Fill a beaker or container with the water to be filtered. Insert the tubing into the container. Slowly draw the water into the syringe by pulling up on the syringe plunger



3. Draw about 50 mL of sample into the syringe. Slowly push on the plunger to force the water through the filter and into a graduated cylinder or volumetric flask. Repeat Steps 2 and 3 until the desired amount of water is obtained.

Note: *As the filter clogs, it gets more difficult to push water through it. At this point, discard the filter and attach a new filter. Replacement filters are available in packages of 10 (Cat. No. 23238-10)*

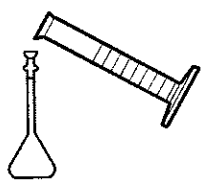
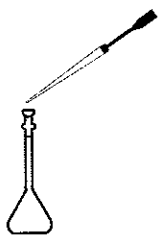
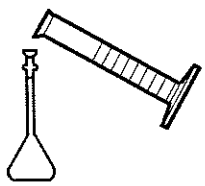
3.6.2.4 Preparing Formazin Dilutions (Factory recommended)

Hach Company recommends using 20, 100, and 800 NTU formazin standards for calibrating the 2100P Turbidimeter. Dilutions with other NTU values can be prepared and used (see *Section 3.6.3.1* on page *38*). If problems occur when using alternate solutions, use the dilutions specified here.

Prepare all formazin dilutions immediately before use and discard after calibration. The 4000 NTU solution is stable for up to a year, but dilutions deteriorate more rapidly. Use the same high quality water (turbidity <0.5 NTU) for the dilutions and the blank

Preparing the 20, 100 and 800 NTU standards

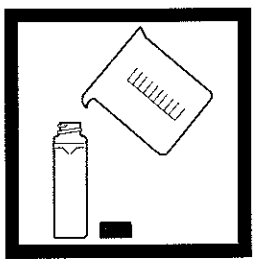
Table 1 Formazin Standard Preparation

	Step 1	Step 2	Step 3
Standards			
20 NTU	Add 100 mL of dilution water to a clean **200-mL** class A volumetric flask.	With a TenSette* pipet, add 1 00 mL of well-mixed 4000 NTU Formazin stock solution to the 200-mL flask.	Dilute to the mark with dilution water Stopper and mix
100 NTU	Add 100 mL of dilution water to a clean **200-mL** class A volumetric flask	With a TenSette pipet, add 5 00 mL of well-mixed 4000 NTU Formazin stock solution to the 200-mL flask.	Dilute to the mark with dilution water Stopper and mix
800 NTU	Add 50 mL of dilution water to a clean **100-mL** class A volumetric flask	With a TenSette pipet, add 20.00 mL of well-mixed 4000 NTU Formazin stock solution to the 100-mL flask.	Dilute to the mark with dilution water. Stopper and mix.

* A class A volumetric pipet may be used in place of a TenSette Pipet.

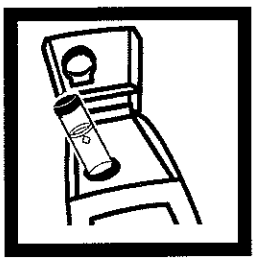
3.6.3 Calibrating the Turbidimeter

Note: *For best accuracy use the same sample cell or four matched sample cells for all measurements during calibration. Always insert the cell so the orientation mark placed on the cell during the matching procedure is correctly aligned. (See Section 2.3.4 on page 15 for matching sample cells).* *



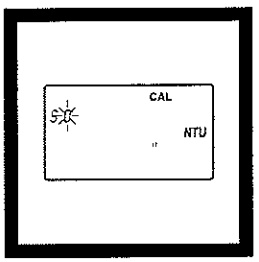
1. Rinse a clean sample cell with dilution water several times. Then fill the cell to the line (about 15 mL) with dilution water or use StablCal <0.1 NTU standard.

Note: *The same dilution water used for preparing the standards must be used in this step.*



2. Insert the sample cell in the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid. Press **I/O**.

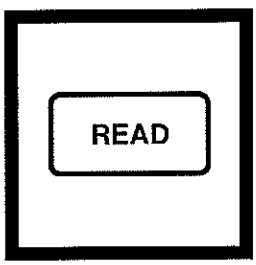
Note: *Choose signal average mode option (on or off) before pressing* **CAL** *– the* **SIGNAL AVERAGE** *key is not functional in calibration mode*



3. Press: **CAL**

The **CAL** and **S0** icons will be displayed (the **0** will flash). The 4-digit display will show the value of the S0 standard for the previous calibration. If the blank value was forced to 0.0, the display will be blank (as shown). Press →to get a numerical display.

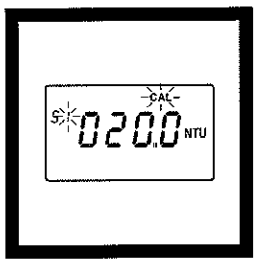
Hach Company only recommends the use of StablCal® Stabilized Formazin or formazin standards for the calibration of Hach turbidimeters. Hach Company cannot guarantee the performance of the turbidimeter if calibrated with co-polymer styrene divinylbenzene beads or other suspensions.



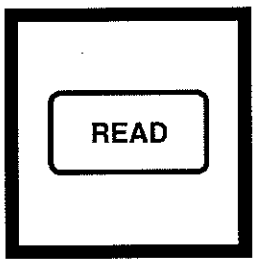
4. Press: **READ**

The instrument will count from 60 to 0, (67 to 0 if signal average is on), read the blank and use it to calculate a correction factor for the 20 NTU standard measurement. If the dilution water is ≥ 0 5 NTU, E 1 will appear when the calibration is calculated (See *Section 3 6 2.3* on page *31* for more dilution water information) The display will automatically increment to the next standard. Remove the sample cell from the cell compartment.

Note: *The turbidity of the dilution water can be "forced" to zero by pressing →rather than reading the dilution water. The display will show* **S0 NTU** *and the ↑ key must be pressed to continue with the next standard*



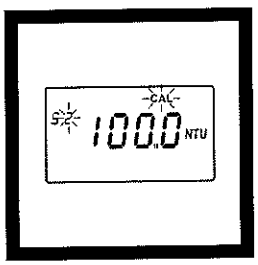
5. The display will show the **S1** (with the 1 flashing) and **20 NTU** or the value of the S1 standard for the previous calibration If the value is incorrect, edit the value by pressing the →key until the number that needs editing flashes Use the ↑ key to scroll to the correct number. After editing, fill a clean sample cell to the line with **well mixed** 20 NTU StablCal Standard or 20 NTU formazin standard. Insert the sample cell into the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid

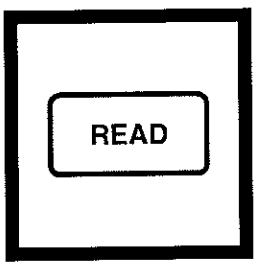


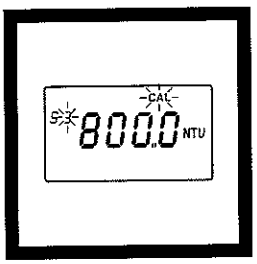
6. Press: **READ**

The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value The display will automatically increment to the next standard Remove the sample cell from the cell compartment

*





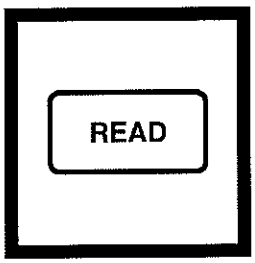


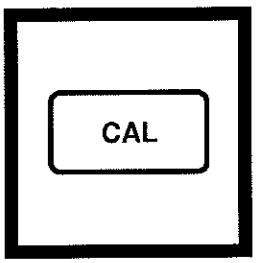
7. The display will show the **S2** (with the 2 flashing) and **100 NTU** or the value of the S2 standard for the previous calibration. If the value is incorrect, edit the value by pressing the →key until the number that needs editing flashes. Use the ↑ key to scroll to the correct number. After editing, fill a clean sample cell to the line with **well mixed** 100 NTU StablCal Standard or 100 NTU formazin standard. Insert the sample cell into the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid.

8. Press: **READ**

The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. Then, the display will automatically increment to the next standard. Remove the sample cell from the cell compartment.

9. The display will show the **S3** (with the 3 flashing) and **800 NTU** or the value of the S3 standard for the previous calibration. If the value is incorrect, edit the value by pressing the →key until the number that needs editing flashes. Use the ↑ key to scroll to the correct number. After editing, fill a clean sample cell to the line with **well mixed** 800 NTU StablCal Standard or 800 NTU formazin standard. Insert the sample cell into the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid.





10. Press: **READ**

The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. Then the display will increment back to the S0 display. Remove the sample cell from the cell compartment

11. Press: **CAL** to accept the calibration. The instrument will return to measurement mode automatically

Note: *Pressing* **CAL** *completes the calculation of the calibration coefficients. If calibration errors occurred during calibration, error messages will appear after* **CAL** *is pressed. If* **E 1** *or* **E 2** *appear, check the standard preparation and review the calibration, repeat the calibration if necessary. If* **CAL?** *appears, an error may have occurred during calibration. If* **CAL?** *is flashing, the instrument is using the default calibration*

NOTES

- If the **I/O** key is pressed during calibration, the new calibration data is lost and the old calibration will be used for measurements. Once in calibration mode, only the **READ**, **I/O**, ↑ , and →keys function Signal averaging and range mode must be selected before entering the calibration mode.

- If **E 1** or **E 2** are displayed, an error occurred during calibration. Check the standard preparation and review the calibration; repeat the calibration if necessary Press **DIAG** to cancel the error message (**E 1** or **E 2**) To continue without repeating the calibration, press **I/O** twice to restore the previous calibration. If **CAL?** is displayed, an error may have occurred during calibration The previous calibration may not be restored. Either recalibrate or use the calibration as is

- To review a calibration, press **CAL** and then ↑ to view the calibration standard values. As long as **READ** is never pressed and **CAL** is not flashing, the calibration will not be updated. Press **CAL** again to return to the measurement mode

3.6.3.1 Preparing User-selected Formazin Dilutions

The formazin solutions should span the entire range of the instrument. Hach recommends preparing three standards:

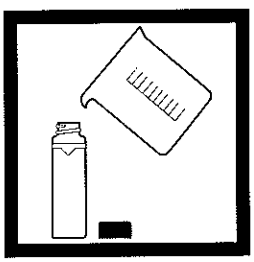
1. 10 to 30 NTU
2. 90 to 110 NTU
3. 700 to 900 NTU

The standards must have a difference of at least 60 NTU.
In addition, a blank made from the dilution water should be prepared.

Prepare the formazin standard solutions from the well mixed 4000 NTU stock solution as specified in *Section 3.6.2.4* on page *32* and dilution water as specified in *Section 3.6.2.2* and *Section 3.6.2.3* on page *31*. Make the standards **immediately** before use and discard them after calibration is done

3.6.3.2 Calibrating with User-selected Standards

Note: *For best accuracy use the same sample cell or four matched sample cells for all measurements during calibration Always insert the sample cell with the same orientation.*



1. Fill a clean sample cell to the line (about 15 mL) with dilution water

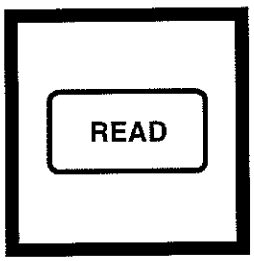
Note: *The same dilution water used for preparing the standards must be used in this step.*

2. Insert the sample cell into the cell compartment and close the lid. Press **I/O**.

Note: *Choose signal average mode option (on or off) before pressing* **CAL** *– the* **SIGNAL AVERAGE** *key is not functional in calibration mode*

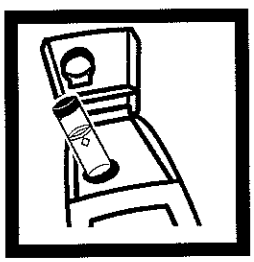
3. Press: **CAL**.

The **CAL** and **S0** icons will appear (the **0** will flash). The 4-digit display will show the value of the S0 standard for the previous calibration

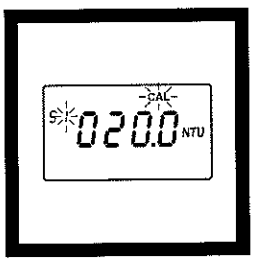


4. Press: **READ**.

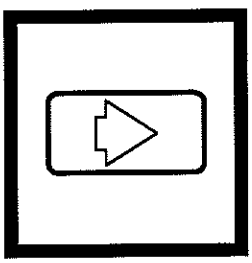
The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the blank and use it to calculate a correction factor for the lowest standard. If the dilution water is ≥0 5 NTU, **E 1** will appear (see *Section 3 6 2 3* on page *31* for more dilution water information). The display will automatically increment to the next standard Remove the sample cell from the cell compartment.



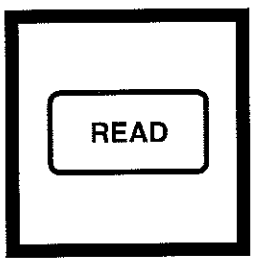
5. Thoroughly mix the 10 to 30 NTU range standard, then fill a clean sample cell to the line with the standard Insert the sample cell into the cell compartment



6. The display will show the **S1** icon (with the **1** flashing) and **20 NTU** or the value of the S1 standard for the previous calibration

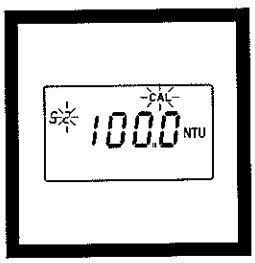


7. Edit the standard concentration by pressing → The **1** will stop flashing and the left digit in the display will flash Press ↑ to scroll the digit up to the appropriate number Press →again to move the cursor to the next digit and edit it in the same manner.

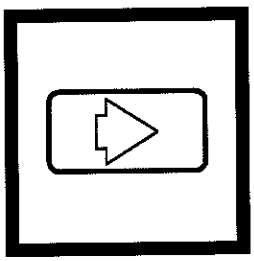


8. When all the digits show the appropriate value, press **READ**. The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value The display will automatically increment to the next standard. Remove the sample cell from the cell compartment

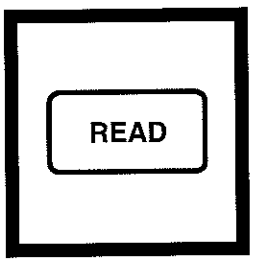
9. Thoroughly mix the 90 to 110 NTU standard, then fill a clean sample cell to the line with the standard. Insert the cell into the cell compartment.



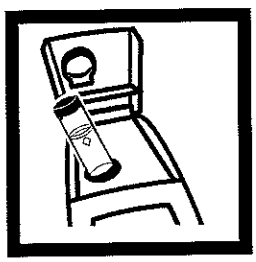
10. The display will show the **S2** icon (with the **2** flashing) and **100 NTU** or the value of the S2 standard for the previous calibration



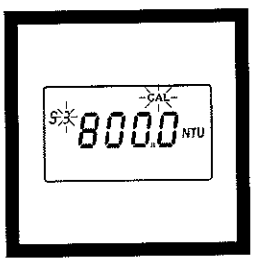
11. Edit the standard concentration by pressing → The **2** will stop flashing and the left digit in the display will flash. Press ↑ to scroll the digit up to the appropriate number Press →again to move the cursor to the next digit and edit it in the same manner



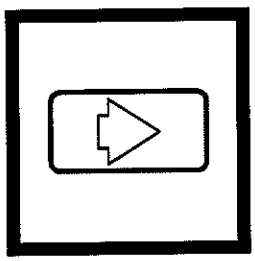
12. When all the digits show the appropriate value, press **READ** The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. Remove the sample cell from the cell compartment



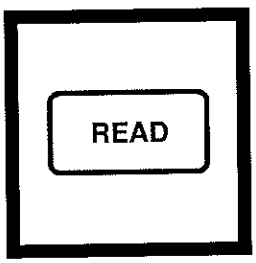
13. Thoroughly mix the 700 to 900 NTU standard, then fill a clean sample cell to the line with the standard Insert the cell into the cell compartment

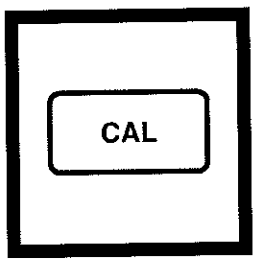


14. The display will show the **S3** icon (with the **3** flashing) and **800 NTU** or the value of the S3 standard for the previous



15. Edit the standard concentration by pressing → The **3** will stop flashing and the left digit in the display will flash Press ↑ to scroll the digit up to the appropriate number Press →again to move the cursor to the next digit and edit it in the same manner.





16. When all the digits show the appropriate value, press **READ**
The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. The instrument will increment back to **S0** Remove the sample cell from the cell compartment

17. Press: **CAL**

The instrument will store the new calibration data and return the instrument to the measurement mode. It will use the new calibration to calculate turbidity for subsequent measurements.

Note: *Pressing* **CAL** *completes the calculation of the calibration coefficients If calibration errors occurred during calibration, error messages will appear after* **CAL** *is pressed If* **E 1** *or* **E 2** *appear, check the standard preparation and review the calibration; repeat the calibration if necessary. If* **CAL?** *appears, an error may have occurred during calibration. If* **CAL?** *is flashing, the instrument is using the default calibration*

NOTES

- If the **I/O** key is pressed during calibration, the new calibration data is lost and the old calibration will be used for measurements. Once in calibration mode, only the **READ, I/O,** ↑ , and →keys function. Signal averaging and range mode must be selected before entering the calibration mode

- If **E 1** or **E 2** are displayed, an error occurred during calibration. Check the standard preparation and review the calibration; repeat the calibration if necessary. If the error messages recur, calibrate using the factory specified standards, *Section 3.6.2 4* on page *32* and *Section 3.6.3* on page *34*. Press **DIAG** to cancel the error message (**E 1** or **E 2**). To continue without repeating the calibration, press **I/O** twice to restore the previous calibration. If **CAL?** is displayed, an error may have occurred during calibration. The previous calibration may not be restored. Either recalibrate or use the calibration as is.

- To review a calibration, press **CAL** and then only ↑ to view the calibration standard values. As long as **READ** is never pressed and **CAL** isn't flashing, the calibration will not be updated. Press **CAL** again to return to the measurement mode.

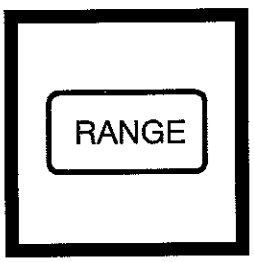
3.6.4 Using Gelex® Secondary Turbidity Standards

Note: *Store Gelex standards at room temperature. Do not allow to freeze or exceed 50 °C.*

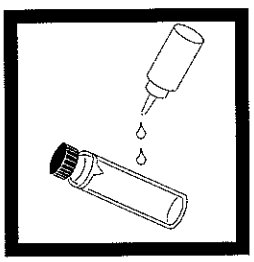
The instrument comes with Gelex Secondary Standards which are particulate suspensions similar to formazin primary standards in light scattering characteristics. NTU values on the Gelex standards indicate the range for which they should be used. Due to minor variations in glass and individual instrument optical systems, the true value of the Gelex standards must be determined against formazin in the same instrument they will be used with for later calibration checks.

3.6.4.1 Assigning Values to Gelex Standards

1. Calibrate the instrument with formazin.



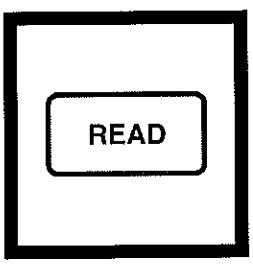
2. Select automatic range mode using the **RANGE** key.



3. Thoroughly clean the outside of the Gelex vials and apply a thin coating of silicone oil

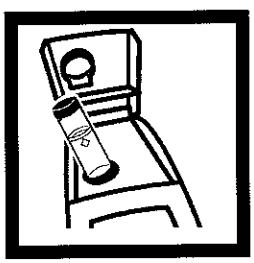
4. Place the 0-10 NTU Gelex standard in the cell compartment so the diamond on the vial aligns with the orientation mark on the instrument. Close the sample lid.

Note: *Correct cell orientation is essential to obtain accurate Gelex values. Always orient the cell so the diamond mark aligns with the orientation mark on the instrument.*



5. Press: **READ**

Record the displayed value, remove the vial from the instrument and mark the value on the band near the top of the vial.



6. Repeat *step 3* through *step 5* for the other Gelex standards, being careful to orient the cells properly.

Re-assign with every formazin calibration

7. Re-assign values to the Gelex standards each time the instrument is calibrated with formazin.

3.6.4.2 Routine Calibration Check With Gelex Standards

The 2100P Turbidimeter does not require standardization before every measurement as some turbidimeters do. Periodically, as experience dictates, check the instrument calibration using the appropriate Gelex Secondary Standard. Be sure the Gelex standards are aligned correctly when inserting them (diamond aligns with orientation mark). If the reading is not within 5% of the previously established value, recalibrate the instrument with StablCal Stabilized Formazin Primary Standard or formazin primary standard (*Section 3.6.3* on page *34*).

MAINTENANCE

Some of the following manual sections contain information in the form of warnings, cautions and notes that require special attention. Read and follow these instructions carefully to avoid personal injury and damage to the instrument. Only personnel qualified to do so, should conduct the maintenance tasks described in this portion of the manual.

Certains des chapitres suivants de ce mode d'emploi contiennent des informations sous la forme d'avertissements, messages de prudence et notes qui demandent une attention particulière. Lire et suivre ces instructions attentivement pour éviter les risques de blessures des personnes et de détoriation de l'appareil. Les tâches d'entretien décrites dans cette partie du mode d'emploi doivent être seulement effectuées par le personnel qualifié pour le faire.

Algunos de los capítulos del manual que presentamos contienen muy importante información en forma de alertas, notas y precauciones a tomar. Lea y siga cuidadosamente estas instrucciones a fin de evitar accidentes personales y daños al instrumento. Las tareas de mantenimiento descritas en la presente sección deberán ser efectuadas únicamente por personas debidamente cualificadas.

Einige der folgenden Abschnitte dieses Handbuchs enthalten Informationen in Form von Warnungen, Vorsichtsmaßnahmen oder Anmerkungen, die besonders beachtet werden müssen. Lesen und befolgen Sie diese Instruktionen aufmerksam, um Verletzungen von Personen oder Schäden am Gerät zu vyyyermeiden. In diesem Abschnitt beschriebene Wartungsaufgaben dürfen nur von qualifiziertem Personal durchgeführt werden.

Algumas das seguintes secções do manual contêm informações em forma de advertências, precauções e notas que requerem especial atenção. Leia e siga atentamente as presentes instruções para evitar ferimentos pessoais e não danificar o instrumento. As tarefas de manutenção descritas nesta parte do manual só poderão ser executadas por pessoal qualificado.

SECTION 4 MAINTENANCE

4.1 Cleaning

Keep the turbidimeter and accessories as clean as possible and store the instrument in the carrying case when not in use. Avoid prolonged exposure to sunlight and ultraviolet light. Wipe spills up promptly Wash sample cells with non-abrasive laboratory detergent, rinse with distilled or demineralized water, and air dry. Avoid scratching the cells and wipe all moisture and fingerprints off the cells before inserting them into the instrument. Failure to do so can give inaccurate readings See *Section 2.3.1* on page *11* for more information about sample cell care

4.2 Battery Replacement

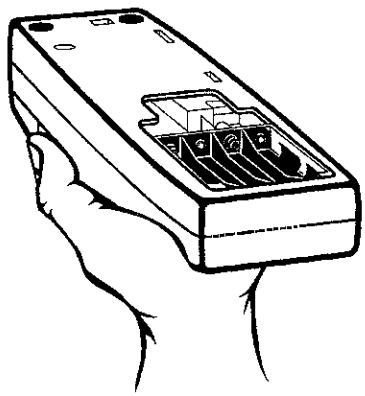
AA alkaline cells typically last for about 300 tests with the signal averaging mode off, about 180 tests if signal averaging is used. The "battery" icon flashes when battery replacement is needed. Refer to *Section 1.4.2* on page *5* for battery installation instructions. If the batteries are changed within 30 seconds, the instrument retains the latest range and signal average selections. If it takes more than 30 seconds, the instrument uses the default settings.

If, after changing batteries, the instrument will not turn off or on and the batteries are good, remove the batteries and reinstall them. If the instrument still won't function, contact Hach Service or the nearest authorized dealer.

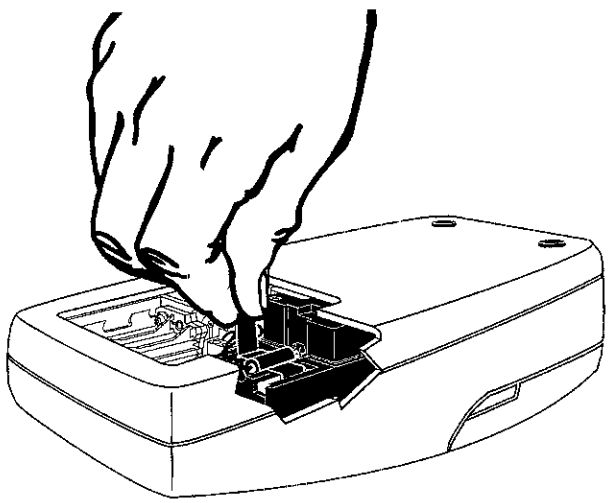
4.3 Lamp Replacement

The procedure below explains lamp installation and electrical connections. Use a small screwdriver to remove and install the lamp leads in the terminal block. The instrument requires calibration after lamp replacement

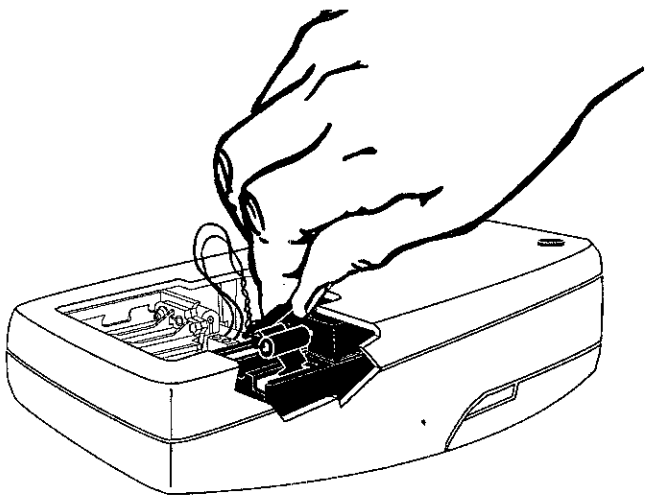
1. Orient the instrument so it is upside down and the top faces away from you. Remove the battery cover and at least one battery.



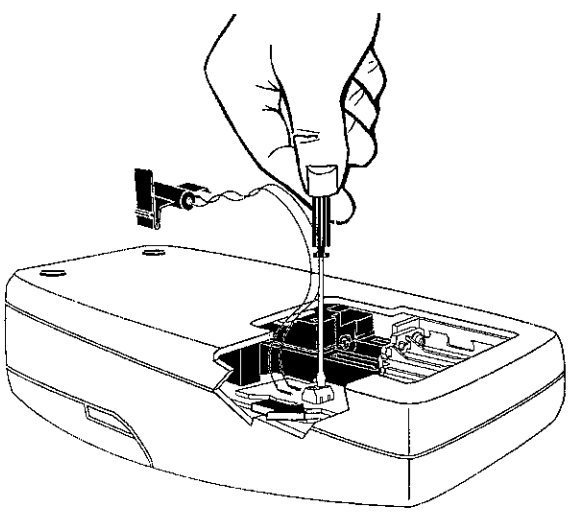
2. Remove the lamp assembly by grasping the tab on the left side of the assembly. Firmly, but gently, slide the assembly towards the rear of the instrument.



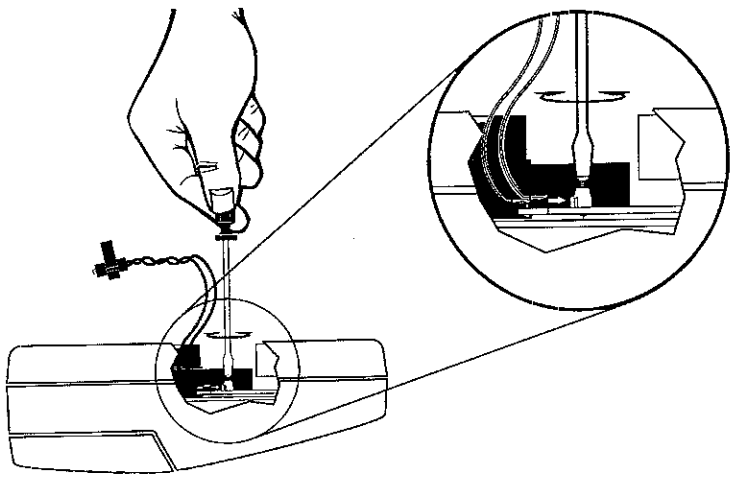
3. Rotate the tab towards the nearest outside edge The assembly should release and slip out easily.



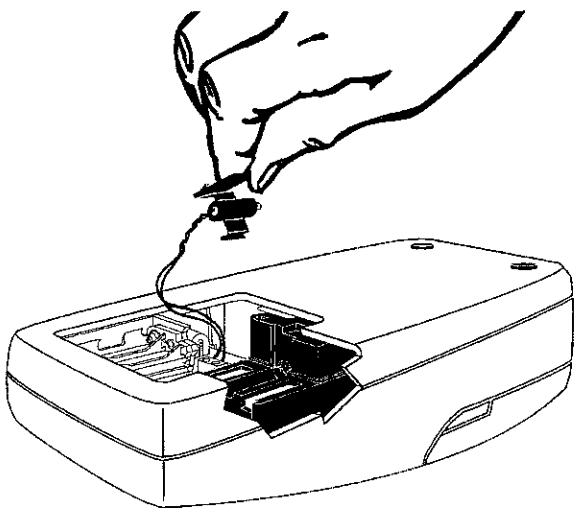
4. Back the terminal block screws **partially** out (1 to 2 turns) and remove the old lamp leads



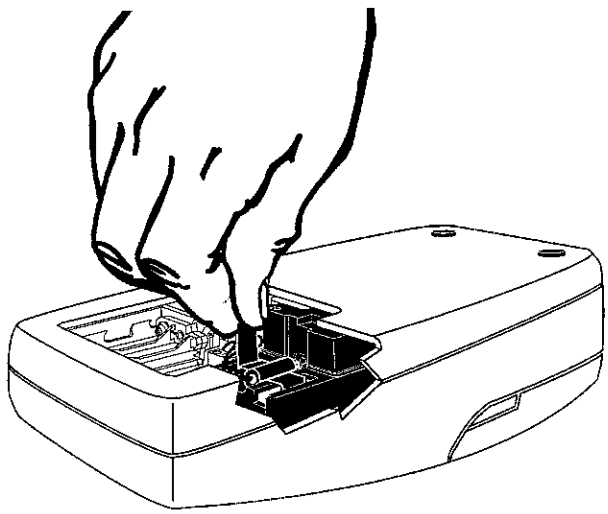
5. Gently bend the wires of the new lamp assembly into an "L" shape so they fit easily into the housing Insert the leads into the terminal screws and tighten with clockwise turns Gently tug on the wires to make sure they are connected to the terminal block



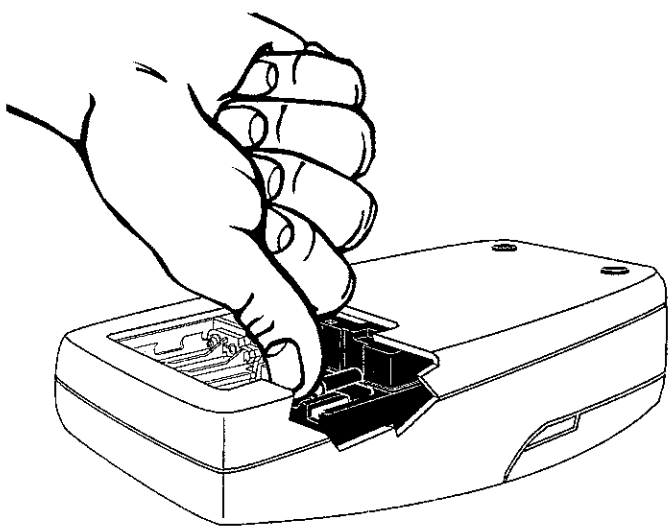
6. Hold the new lamp assembly by the tab with the lamp facing the tope (keyboard) of the instrument Slide the small catch on the other side of the assembly into the black plastic slot (towards the nearest edge of the instrument)



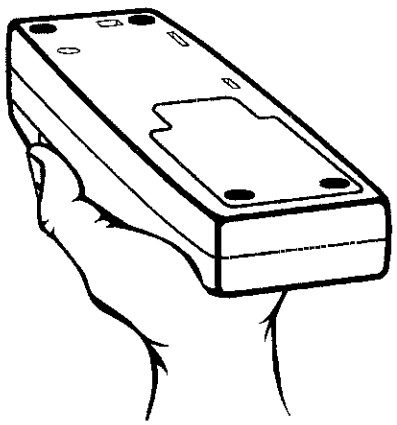
7. Snap the U-shaped bottom of the tab into the slot on the left side of the black plastic that holds the lamp assembly



8. With your thumb, firmly slide the assembly forward until it stops. Again, push firmly against the tab to make sure the lamp is seated correctly.

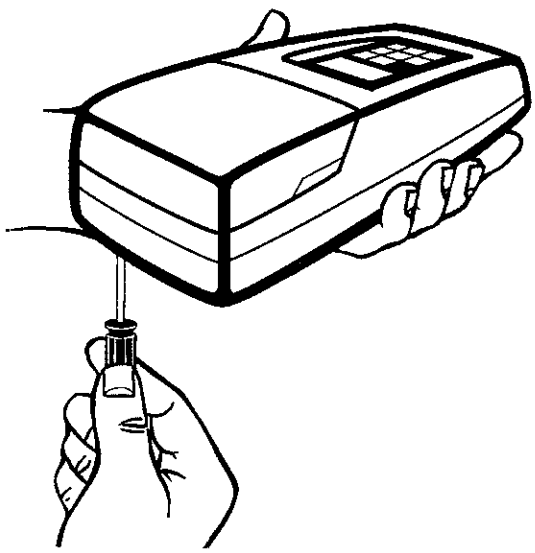


9. Replace the battery(s) and battery cover



10. Insert the 800 NTU formazin standard into the sample cell. Press and hold **READ**. Then press **I/O**. Release the **READ** key after the software version number disappears from the display (for models with serial numbers less than 920300000800, **2100** disappears).

11. Adjust the scattered light amplifier output by inserting a small flat-bladed screwdriver into the trimpot hole (located on bottom). Adjust the display to read 2.5 ± 0.3 volts (2.0 volts for models that display **2100** when turned on).



12. Press **I/O** to exit gain adjust mode.

13. Perform a formazin calibration according to *Section 3.6.3* on page *34* or *Section 3.6.3.1* on page *38*

SECTION 5 TROUBLESHOOTING

5.1 Using the Diagnostic Functions Key

Enter the diagnostic mode by pressing the **DIAG** key. Exit this mode at any time by pressing the key again. The diagnostic mode allows access to information about instrument function which may be useful for servicing and troubleshooting.

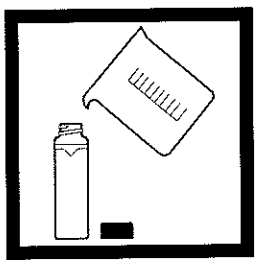
5.1.1 Basic Diagnostic Codes

The diagnostic codes are:

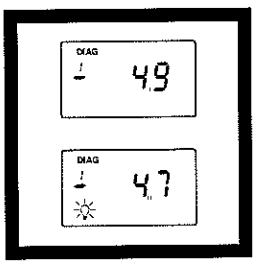
Code	Description
1	Checks the battery voltage with the lamp on, then with the lamp off. This is a dual diagnostic code.
2	Displays calibration coefficient a_0
3	Displays calibration coefficient a_1
4	Displays calibration coefficient b_0
5	Displays calibration coefficient b_1
6	Displays the lamp voltage (about 3 volts)
7	Displays the dark voltage of the transmitted light detector amplifier with the lamp off and the detector amplifier voltage with the lamp on.
8	Displays the high gain dark voltage of the 90° detector amplifier with the lamp off and the detector amplifier voltage with the lamp on.*
9	Displays the low gain dark voltage of the 90° detector amplifier with the lamp off and the detector amplifier voltage with the lamp on.

* Samples with turbidity >10 NTU may display - - - for the lamp-on amplifier voltage.

5.2 The Diagnostic Procedure

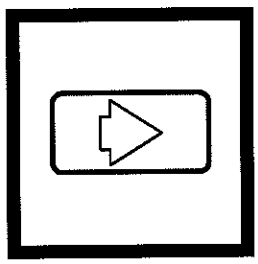


1. Fill a clean sample cell to the line with clear water, cap the cell and place it in the cell compartment Press the **READ** key and wait until the reading is finished.

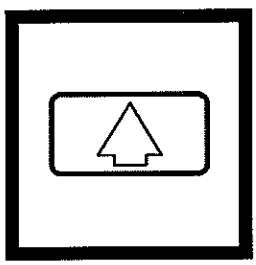


2. Press: **DIAG**

The **DIAG** icon will turn on and **1** will be displayed below the icon The instrument will measure the battery voltage with the lamp off and display the result in volts (V). Then the lamp icon will turn on and the instrument will measure the voltage with the lamp on. The value is briefly displayed before defaulting to the lamp-off reading. To repeat the measurement, press **READ**.



3. To continuously display the lamp-on voltage, press → The lamp icon will flash Press →to turn the lamp icon off (the lamp is not on during this display).



4. Press the ↑ key to scroll through the other diagnostics. Each press of the key increments the digit in the small numerical display below the **DIAG** icon and the result of the diagnostic measurement is then displayed. Each press of the **READ** key updates the value. For measurements made with the lamp off and again with the lamp on, the measurement with the lamp off is displayed when the diagnostic is entered. To see the second measurement with the lamp on, press the →key (only works with diagnostic codes 1, 7, 8, & 9). The lamp icon will flash and the lamp-on measurement will be displayed in volts. Press →to turn the lamp icon off.

Note: *DIAG 8 will display ---- for the lamp-on voltage if a of >10 NTU is placed in the cell compartment*

5.3 Other Instrument Diagnostics

5.3.1 Display Test

Pressing and holding the I/O key turns on all the display icons and elements so you can determine if all the elements and icons are functioning. The display test sequence will cycle as long as the key is held down.

5.4 Error Messages

Error messages indicate sample interferences and/or instrument malfunction.

5.4.1 Flashing Numeric Display

If the highest value in the range selected is flashing in the display, the sample is too turbid (or overrange) for the selected range. In automatic or manual range, **1000** flashes if the sample is over the instrument's range. In manual range mode, select the next higher range mode if **9.99** or **99.9** flashes. See *Section 2.3.6* on page *21* for measuring overrange samples. The display will stop flashing if a sample within range is inserted and read.

5.4.2 E Messages

An error message indicates either an instrument failure or an operation cannot be performed. **An error message can be cleared by pressing DIAG** (display will return to previous measurement or calibration value). The meter continues to operate as best it can. If the message occurs during a calibration, calibration can continue. If the error message occurs when a calibration is being calculated, the instrument will discard the new calibration and retain the old calibration. Error messages and corrective actions are listed below.

5.4.3 CAL?

A flashing **CAL?** appears when the instrument is using the default calibration programmed at the factory. It will appear if the analyst has erased the user-entered calibration using the procedure to restore the default calibration or after and E 4 error is cleared by pressing DIAG. Recalibrate as soon as possible when **CAL?** appears. **CAL?** (not flashing) appears when a calibration has questionable validity.

Message*	Probable Cause	Corrective Action
E1	Dilution water is ≥ 0.5 NTU.	Start calibration over with better quality dilution water or use a membrane filter to filter the water before use.
E2	Two standards have the same value or their difference is less than 60 NTU. Not all standards were read during the calibration Standard 1 is too low (<10 NTU).	Recheck preparation of standards and repeat calibration
E3	Low light error	Re-read measurement. Check lamp** Check for obstructed light path. Dilution may be necessary
E4	EEPROM malfunction.	Check sum failed Press **I/O** If E 4 reappears, call Hach service If **CAL?** appears, recalibrate.
E5	A/D overrange.	Check for obstructed light path. Call Hach Service.
E6	A/D underrange.	Check for open lid during reading and re-read. Check for obstructed light path. If persists, call Hach Service.
E7	Light Leak	Close lid before pressing **READ** key.
E8	Bad lamp circuit	Reinsert lamp leads at terminal block-make sure the lead ends are not touching each other.If this fails, call Hach Service.

* Error messages 4, 5, and 6 may indicate a failure in the internal electronics
** Check lamp by inserting a pencil or piece of paper into the cell compartment and pressing READ Light should be visible on the inserted object.

GENERAL INFORMATION

At Hach Company, customer service is an important part of every product we make.

With that in mind, we have compiled the following information for your convenience.

REPLACEMENT PARTS & ACCESSORIES

REPLACEMENT PARTS

Description	Cat. No.
<0.1 NTU* StablCal®** Stabilized Formazin Standard, 100 mL	26597-42
20 NTU StablCal® Stabilized Formazin Standard, 100 mL	26601-42
100 NTU StablCal® Stabilized Formazin Standard, 100 mL	26602-42
800 NTU StablCal® Stabilized Formazin Standard, 100 mL	26605-42
AA Batteries, 4/pkg	19380-04
Battery Door	46005-00
Carrying Case	46506-00
Gelex® Standards, set (includes standards and 3 sample cells)	24641-05
Instrument Manual	46500-88
Lamp Assembly, with leads	46539-00
Mounting Feet, 4/pkg	41093-00
Oiling Cloth	47076-00
Sample Cells, 1 inch, with cap, 6/pkg	24347-06
Silicone Oil, 15 mL	1269-36

OPTIONAL ACCESSORIES AND REAGENTS

Deionized Water, 3.78 L	272-17
Bath, Ultrasonic, 2.8 L (0.75-gal), w/heater	24895-00
Battery Charger, 120 V	46479-00
Battery Charger, 230 V	46479-01
Battery Eliminator, 120 V	46079-00
Battery Eliminator, 230 V	46080-00
Filter, 0.2 micron, 10/pkg	23238-10
Formazin, 4000 NTU, 500 mL	2461-49
Formazin, 4000 NTU,100 mL	2461-42
Hexamethylenetetramine, 100 g	1878-26
Hexamethylenetetramine, 500 g	1878-34
Hydrazine Sulfate, 20 g	742-46
Hydrazine Sulfate, 100 g	742-26
NiCad Rechargeable Battery (4 required)	16077-00
Pipet, serologic, 1.00 mL	532-35

* <0.1 NTU StablCal® Standard is used in place of dilution water standard when performing a calibration

** StablCal® is a registered trademark of Hach Company

OPTIONAL ACCESSORIES AND REAGENTS, continued

Description	Cat. No.
Pipet, TenSette®*, 1-10 mL	19700-10
Pipet Tips, for 1-10 mL TenSette Pipet, 50/pkg	21997-96
Pipet Tips, for 1-10 mL TenSette Pipet, 1000/pkg	21997-28
Pipet, Volumetric, Class A, 1.00 mL	14515-35
Pipet, Volumetric, Class A, 5.00 mL	14515-37
Pump, Vacuum, Hand-Operated	14283-00
Pump, Vacuum, 115 V, 60 Hz	14697-00
Pump, Vacuum, 230 V, 50 Hz	14697-02
Sample Degassing Kit	43975-00
Sample Filtration and Degassing Kit	43975-10
StablCal® Low Level Turbidity Standards for Instrument Verification**	
0.1 NTU, 100 mL	27233-42
0.3 NTU, 100 mL	26979-43
0.5 NTU, 100 mL	26980-42
StablCal® Calibration Set for 2100P Turbidimeter	
<0.1, 20, 100, 800 NTU, 500 mL each	26594-00
<0.1, 20, 100, 800 NTU, 100 mL each	26594-10
<0.1, 20, 100, 800 NTU, sealed vials	26594-05
Triton-X Solution, 118 mL (4 oz)	14096-32
Volumetric Flask, 100 mL	14574-42
Volumetric Flask, 200 mL	14574-45

* TenSette™ is a Hach Company trademark.

** Do not use these standards for instrument calibration.

HOW TO ORDER

By Telephone:
6:30 a m to 5:00 p m MST
Monday through Friday
(800) 227-HACH
(800-227-4224)
By FAX:
(970) 669-2932

By Mail:
Hach Company
P.O. Box 389
Loveland, Colorado 80539-0389
U.S.A
Ordering information by E-mail:
orders@hach.com

Information Required

- Hach account number (if available)
- Your name and phone number
- Purchase order number
- Brief description or model number
- Billing address
- Shipping address
- Catalog number
- Quantity

Technical and Customer Service (U.S.A. only)

Hach Technical and Customer Service Department personnel are eager to answer questions about our products and their use Specialists in analytical methods, they are happy to put their talents to work for you. Call **1-800-227-4224** or E-mail **techhelp@hach.com**

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In Latin America, the Caribbean, the Far East,
the Indian Subcontinent, Africa, or the Pacific Basin:
Hach Company World Headquarters; Loveland, Colorado, U.S A.
Telephone: (970) 669-3050; FAX: (970) 669-2932

REPAIR SERVICE

Authorization must be obtained from Hach Company before sending any items for repair. Please contact the Hach Service Center serving your location

In the United States:
Hach Company
100 Dayton Ave
Ames, Iowa 50010
(800) 227-4224 (U.S.A. only)
Telephone: (515) 232-2533
FAX: (515) 232-1276

In Canada:
Hach Sales & Service Canada Ltd.
1313 Border Street, Unit 34
Winnipeg, Manitoba
R3H 0X4
(800) 665-7635 (Canada only)
Telephone: (204) 632-5598
FAX: (204) 694-5134

In Latin America, the Caribbean, the Far East, the Indian Subcontinent, Africa (except Mediterranean Africa) or Basin:
Hach Company World Headquarters,
P.O. Box 389
Loveland, Colorado 80539-0389 U.S.A.
Telephone: (970) 669-3050
FAX: (970) 669-2932

In Europe, the Middle East, or in Mediterranean Africa:
Hach Europe, S.A./N.V.
Chaussée de Namur, 1
B-5150 Floriffoux (Namur), Belgium
Telephone: (32)(81) 44 71 71
FAX: (32)(81) 44.13.00

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Hach warrants most products against defective materials or workmanship for at least one year from the date of shipment; longer warranties may apply to some items.

HACH WARRANTS TO THE ORIGINAL BUYER THAT HACH PRODUCTS WILL CONFORM TO ANY EXPRESS WRITTEN WARRANTY GIVEN BY HACH TO THE BUYER. EXCEPT AS EXPRESSLY SET FORTH IN THE PRECEDING SENTENCE, HACH MAKES NO WARRANTY OF ANY KIND WHATSOEVER WITH RESPECT TO ANY PRODUCTS. HACH EXPRESSLY DISCLAIMS ANY WARRANTIES IMPLIED BY LAW, INCLUDING BUT NOT LIMITED TO ANY WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE.

LIMITATION OF REMEDIES: Hach shall, at its option, replace or repair nonconforming products or refund all amounts paid by the buyer **THIS IS THE EXCLUSIVE REMEDY FOR ANY BREACH OF WARRANTY.**

LIMITATION OF DAMAGES: IN NO EVENT SHALL HACH BE LIABLE FOR ANY INCIDENTAL OR CONSEQUENTIAL DAMAGES OF ANY KIND FOR BREACH OF ANY WARRANTY, NEGLIGENCE, ON THE BASIS OF STRICT LIABILITY, OR OTHERWISE.

This warranty applies only to Hach products purchased and delivered in the United States.

Catalog descriptions, pictures and specifications, although accurate to the best of our knowledge, are not a guarantee or warranty

For a complete description of Hach Company's warranty policy, request a copy of our Terms and Conditions of Sale for U S. Sales from our Customer Service Department

APPENDIX D
KANSAS CITY DISTRICT DATA QUALITY EVALUATION GUIDANCE

KANSAS CITY DISTRICT DATA QUALITY EVALUATION GUIDANCE
August 18, 2003
Revised February 22, 2006

1 Introduction

This guidance, when used, is meant to be used according to the directions in the project's QAPP for evaluating the quality of the data and will be appended to the QAPP. The QAPP may use any part or none of this guidance as necessary to meet project demands. This is not a laboratory guidance and is not meant to interfere with the laboratory's compliance with the DOD QSM. It is to be used solely by data quality evaluators, reviewers, and usability assessors. This guidance is meant to serve as an initial base from which to build a data quality evaluation program on data generated from a project. When used, it is understood that deviations from the written guidance will sometimes be necessary. It is expected that these deviations will be listed and the deviation(s) justified in writing.

When data fails to conform to the requirements stated in the QAPP (e.g., failure to perform corrective actions), the data stands the risk of being rejected by the data evaluator.

2 Evaluation Procedure

Data quality will be determined by the evaluation of the minimum following items:

- holding times
- method detection limits
- blanks
- laboratory control samples
- matrix spikes/matrix spike duplicates
- matrix duplicates/precision
- surrogate recoveries
- second column confirmation
- internal standards (optional)

2.1 Holding Times

1. In general, exceedence of holding times will be qualified as shown:
   a. If no evidence samples were properly preserved for volatiles, exceedence of a 7-day holding time results in "R".
   b. If samples were properly preserved for volatiles, exceedence of a 14-day holding time results in "R".
   c. For semi-volatile compounds and anions, exceedence of holding time results in "R" qualified data.

2. Deviations to the above must be justified in writing. If comparison with historical data is considered, it must be considered along with other criteria, i.e., historical data comparison will not be the primary reason for acceptance of data (no qualifier).

3. Corrective actions taken after holding time exceedence. Corrective actions that take place after 11/2 times the holding time will be rejected (R). If laboratory routinely corrects after holding time, data will be rejected. Exceptions to this will be justified in writing.

2.2 Method Detection Limits

1. Result falling below either the MDL or the MRL will be U-qualified.
2. Results falling between the MDL/MRL and the PQL will be J-qualified.
3. Unless adequately addressed in the QAPP, data recorded as non-detect will be rejected if detection limit is above the action level (AL).

2.3 Blanks

1. Missing blank and no hierarchy[1] blank available, the following is recommended:
   a. Low positive result of each specific analyte will be U-qualified. Low positive result is defined as any positive number less than 5 times the maximum blank contamination of that analyte found in any blank from any other batch in the dataset.
   b. Medium positive result will be estimated (J). Medium positive result will be any result between the low positive result defined above and 10 times that result.
   c. High positive result will be unqualified.

2. Samples associated with contaminated blanks will be reported with the analytical result followed by "U" when the analytical result is less than 5 times the blank contamination for uncommon lab contaminants or less than 10 times the blanks contamination for common lab contaminants.

2.4 Laboratory Control Samples

1. Missing LCS. Laboratory will be contacted for missing LCS. Otherwise, data will be rejected.
2. For specific chemicals of concern, LCS recoveries must be within criteria. If not, a corrective action must bring them into criteria. If this fails, the following qualifier assignment applies:
   a. For purge-and-trap, recoveries outside lab criteria but within 40-160% or 4σ, whichever results in the wider range, result in J-coding the analyte(s) in all samples in the batch. If outside 40-160% or 4σ, data is R-coded.
   b. For inorganic analyses, recoveries outside lab criteria but within 60-140% or 4σ, whichever results in the wider range, result in J-coding the analyte(s) in all samples in the batch. If outside the 60-140% or 4σ, data is R-coded.
   c. For semivolatiles, recoveries outside lab criteria but within 60-140% or 4σ, whichever results in the wider range, result in J-coding the analyte(s) in all samples in the batch. If outside the 60-140% or 4σ, data is R-coded.

[1] A hierarchy blank is a blank that is used to evaluate contamination resulting from processes preceding and including the subject missing blank. That is, either an equipment blank or trip blank may be used as a hierarchy blank to the method blank.

3. Marginal exceedences[2] will apply to all other chemicals. Chemicals that fail in this category will be rejected. Acceptable recovery ranges for marginal exceedences are listed in item 2 above.
4. When action levels (AL) have been listed, sample results that are greater than 10-fold higher than the AL will be unqualified regardless of the magnitude of the LCS failure. Samples whose results are less than 10-fold different (high or low) will be qualified according to the criteria listed under **Matrix Spikes/Matrix Spike Duplicates.**

2.5 Matrix Spikes/Matrix Spike Duplicates

Paragraphs 3 through 5 evaluate whether it can be determined by the analytical result if the actual concentration of the analyte of concern is above or below the action level (AL). If the analytical result is too close to the action level to make such a determination, the result is rejected as being unusable. Paragraphs 3 through 5 allows the data evaluator to make this determination. Note that these calculations are not needed if it is clear that the analytical result is far removed from the AL.

1. All matrix spikes outside acceptance criteria will be J-coded as a minimum.
2. Qualification will be based on the most significant failure in the MS and MSD pair.
3. For MS recoveries <100%, the analytical result is rejected if the analytical result falls at or below the action level (AL) or at or above the number calculated from $\frac{(R)(AL)(200-RPD)}{100(200+RPD)}$, or AL $\geq$ sample result $\geq \frac{(R)(AL)(200-RPD)}{100(200+RPD)}$.

   Where

   AL = Action Level
   R = Percent Recovery
   RPD = Relative Percent Difference

   For example, if MS recovery (*R)* of chrysene is 75%, its RPD is 20% and its AL = 10 ppb, then data that is equal to or greater than 6.13 ppb or equal to or less than 10 ppb is rejected[3]. If MS recoveries are not available for any specific COC, use LCS.
4. For MS recoveries >100%, the analytical result is rejected if the analytical result falls at or above the action level (AL) or at or below the number calculated from $\frac{(R)(AL)(200+RPD)}{100(200-RPD)}$, or AL $\leq$ sample results $\leq \frac{(R)(AL)(200+RPD)}{100(200-RPD)}$.
5. If the recovery of one member of the MS-MSD pair is <100% and the recovery of the other member is >100%, then sample result is rejected if $\frac{(R_1)(AL)(200-RPD)}{100(200+RPD)} \leq$ sample result

[2] A marginal exceedence is defined as being beyond the LCS control limit (3 standard deviations) but within the exceedence limit (4 standard deviations). The number of chemicals permitted to have MEs depend on the total number of chemicals in the LCS. This number may be found in the DoD QSM, DoD Appendix D, Section D2, page 180.
[3] The formula presented is calculating a concentration that is below the AL by the same amount as the percent MS recovery with an additional lowering considering the analytical variability (expressed as RPD).

$\leq \frac{(R_2)(AL)(200+RPD)}{100(200-RPD)}$ with R_1 and R_2 the recoveries under 100% and over 100%, respectively.

6. Sample results not falling into the above ranges will be rejected if the MS or MSD fails and the LCS recovery falls outside the recoveries mentioned under **Laboratory Control Samples,** item 2.

2.6 Matrix Duplicates/Precision

a. Refer to **Matrix Spikes/Matrix Spike Duplicates** which may be used to cover precision.

b. All other RPDs outside acceptance criteria will be J-coded.

2.7 Surrogate Recoveries

1. If the surrogate is marginally out and the LCS/blanks surrogate is also out, qualify detects of the chemicals considered associated with the non-compliant surrogate (i.e., chemicals with similar RTs and similar structure as surrogate) with “J” and non-detects with “UJ”.
2. If the surrogate is marginally out in the sample and the surrogate in the LCS or blank is in, this should be considered a matrix effect. Qualify chemicals most closely associated with the surrogate(s) with “J”.
3. If the surrogate is grossly outside of 60-140% for purge-and-trap and 20-180% for extractable organics, whether the LCS/blanks surrogates are in or out, a corrective action should have been completed. In the absence of a corrective action, qualify non-detects with “R” and detects with “J”.
4. No qualification if surrogates are out due to a chromatographic problem.
5. No qualification if surrogates are diluted out.
6. Where multiple surrogates are present, if one or more grossly fail, the data is qualified on the most non-compliant surrogate.

2.8 Second Column Confirmation

1. What constitutes acceptable agreement between two columns will be issued by the laboratory.
2. In the case of a peak appearing on primary column, but confirmation not run, the following will apply:
    a. For long term monitoring where the identity of the contamination is known for each well, lack of confirmation will result in a “J” qualifier. If information is critical, a footnote will be given that the location should be resampled;
    b. For areas that have been previously well characterized, and past chromatograms have shown no interferences around the chemicals of concern, lack of confirmation may go unqualified. This basically covers remedial actions; and

c. For areas where contamination is questionable, lack of confirmation will result in a rejection (R) of the data with a footnote that the sampled area requires reinvestigation.

c. When confirmation was attempted, but interferences obscured the peak on the confirmation column, the result would be reported from the column that did not contain interferences and J-qualified. Surrogate recoveries will also be reported from that column.

d. When the RPD between the primary column peak and the secondary column peak is greater than 40%, the quantitation is taken from the smaller peak and J-qualified.

2.9 Internal Standards

1. The internal standard (IS) peak area of the CCV is the standard, acceptable peak area. The IS peak area of samples should be -50% to +100% of this area. IS areas falling outside this range will be qualified according to the following:

    a. For high IS areas in samples which will be greater than 2 times the standard area but less than 5 times the standard area should result in “UJ” for non-detects and “J” for detects.

    b. For IS areas greater than 5 times the standard area, a corrective action would be required. In the absence of a corrective action, any result less than the AL will be rejected. All other results will be J-coded.

    c. For IS areas less than 1/2 the standard area but greater than 1/5 the standard area, positive results will be J-coded.

    d. For IS areas less than 1/5 the standard area in either a matrix spiked sample or the LCS, the matrix spike or LCS recovery respectively will be used to determine any negative impact on the results. For low IS areas found in other samples, the surrogate recoveries will be used for the evaluation. If impacted and the sample result is greater than the AL, the result will be rejected. Result also rejected if not-detected. All other results will be either R or J-coded.

APPENDIX E
GUIDELINES FOR ANALYSIS NOTEBOOK DOCUMENTATION FOR SCREENING SAMPLES

APPENDIX E - GUIDELINES FOR ANALYSIS NOTEBOOK DOCUMENTATION FOR SCREENING SAMPLES

In order to provide full documentation of screening samples a project notebook will be kept, and copies of the relevant pages will faxed to the CENWK project chemist daily and be provided in the data package. The notebook is intended to be a working document and will be kept in real-time as the analysis are performed. Entries into the notebook will be made for each calibration, sample analysis and continuing calibration check so that the process of analysis is traceable. The date and local time will entered into the data system so that date and times on the chromatographic reports are actual.

Project Initiation

At the start of the project the date, time, facility name, site name, sample matrix or matrixes (groundwater, surface soil etc), project objective, company performing the analysis and operator of the instrument will be recorded.

Date
Time
Facility
Site
Sample matrixes
Project objective
Company
Operator

Calibration

A new calibration consisting of at least three concentrations for all analytes will be performed on site before any samples are analyzed. A calibration table will be produced in the project notebook. This table will include all analytes, the elution time of the analyte, the concentration of each standard, the area of each standard, calibration factor and Coefficient of Determination (r^2) for all analytes. The chromatograph report generated by the chromatography software will be included for all calibration standards analyzed.

Analyte	Elution Time	Standard Concentration	Area	Calibration Factor	r^2

If any of the Coefficient of Determination (r^2) is below 0.97 that calibration will be repeated until satisfactory results are obtained for all analytes. Each calibration will be initialed by the analyst indicating acceptance of the results.

Method Blank

A blank will be analyzed prior to sample analysis and repeated daily prior to sample analysis initiation. A table will be made in the project notebook showing the results of the blank analysis and the chromatograph and analysis report will be inserted in the data package for each blank run.

Chromat No.	Time	Analyte	Area	Result

If any analyte is detected in the blank, a second blank will be run. Blanks will continue to be analyzed until the source of contamination is found and all analytes are nondetect.
The analyst will initial the blank table indicating acceptance of the results.

Analysis

Each sample analysis will be recorded in a table that will record the Chromatograph number, time of the analysis, sample ID, sample size, analyte, area and result.
A clearly labeled chromatograph and analysis report generated by the chromatography software will be included for each sample in the data package. Any hand calculations needed to determine the result will be shown on the analysis report. A new table will be started following each continuing calibration check.

Chromat No.	Time	Sample ID	Sample Size	Analyte	Area	Result

Continuing Calibration Checks

Calibration will be checked prior to starting and immediately following an analysis run. Calibration will also be checked every 10 samples throughout the run. Calibration checks will be performed using the middle standard of the standardization. The following information will be recorded in the project notebook for every continuing calibration check; chromatograph number, time, analyte, calibration amount, area, result and percent recovery.

Chromat No.	Time	Analyte	Calibration Amount	Area	Result	% Rec

If the percent recovery is outside of the 75-125% range a second calibration check will be run prior to the

analysis of any sample. If any of the analytes are outside of the 75-125% percent range in the second calibration check the instrument will be recalibrated for all analytes in the analysis that failed before any more samples are analyzed. If the final calibration check is outside the acceptable range a second calibration check will be run. If the second calibration check is outside the acceptable range a new calibration will be performed prior to starting sample analysis for the next analysis run. Each calibration check will be initialed by the analysis indicating acceptance of the result.

An example of a project notebook in proper format has been attached.

1

DATE: 10-Oct-2001
Time: 08:10
Facility: Ft Riley
Site: Abandoned Gas line
matrix: Soil / Ground water
objective: Hot spot Location
Company: Shephard Analytical
operator: T. R. Shephard

Calibration

10-Oct-2001 08:20

CCl4	2.574	1ppb	762417	1.649×10^{-6}	0.9895
		5ppb	3662851		
		10ppb	6198977		
TCE	3.452	1ppb	47337	2.152×10^{-5}	0.9905
		5ppb	267823		
		10ppb	464156		
PCE	5.66	1ppb	112601	8.237×10^{-6}	0.9889
		5ppb	695763		
		10ppb	1200488		

TRS

Method Blank

Chromat #	Time	Analyte	Area	Result
31	11:18	CCl4	0	ND
		TCE	0	ND
		PCE	0	ND

TRS

Analysis

Chromat #	Time	Sample ID	wt.	Analyte	Area	Result
32	11:34	SB-42 (6-8')	5g	CCl4	0	ND
				TCE	0	ND
				PCE	0	ND
33	11:53	SB-43 (6-8')	5g	CCl4	0	ND
				TCE	0	ND
				PCE	0	ND
34	12:10	SB-44 (6-8')	5g	CCl4	0	ND
				TCE	0	ND
				PCE	0	ND
35	12:25	SB-40 (2-4')	5g	CCl4	0	ND
				TCE	0	ND
				PCE	0	ND

1

2

Chromat #	Time	Sample ID	wt	Analyte	AREA	Result
36	12:41	SB-39 (6-8')	5g	CCl4	0	ND
				TCE	0	ND
				PCE	0	ND
37	12:55	SB-41 (6-8')	5g	CCl4	0	ND
				TCE	0	ND
				PCE	0	ND
38	13:18	SB-36 (6-8')	5g	CCl4	0	ND
				TCE	0	ND
				PCE	0	ND
39	13:35	SB-37 (6-8')	5g	CCl4	0	ND
				TCE	0	ND
				PCE	0	ND
40	13:54	SB-38 (6-8')	5g	CCl4	0	ND
				TCE	0	ND
				PCE	0	ND
41	14:07	SB-28 (6-8')	5g	CCl4	0	ND
				TCE	0	ND
				PCE	0	ND

Calibration Check

Chromat #	Time	Analyte	Cal Amount	Area	Result	% Rec
42	14:27	CCl4	5ppb	3020012	4.98	92.6
		TCE	5ppb	177368	3.86	77.2
		PCE	5ppb	645866	5.32	106.4

2nd Calibration Check

Chromat #	Time	Analyte	Cal Amount	Area	Result	% Rec
43	14:36	CCl4	5ppb	3129163	5.16	103.2
		TCE	5ppb	215613	4.64	92.8
		PCE	5ppb	5026 10	4.14	82.8

TRB

* * * * *

APPENDIX F
WORK PLAN

Final Work Plan
OU 1 Remedial Investigation Addendum/Feasibility Study
Former Schilling Air Force Base, Salina Kansas
Burns and McDonnell Engineering Company, Inc., January 12, 2007

(SUBMITTED ON CD)

APPENDIX G
FIELD SAMPLING PLAN

Final Sampling and Analysis Plan
Volume I – Field Sampling Plan,
OU 1 Remedial Investigation Addendum/Feasibility Study
Former Schilling Air Force Base, Salina, Kansas
Burns and McDonnell Engineering Company, Inc., January 12, 2007

(SUBMITTED ON CD)